ARO 30938.1-MS-SB2

TEXAS RESEARCH INSTITUTE AUSTIN, INC.

A Texas Research International Company

ESTIMATING REMAINING LIFE IN BIOLOGICAL/CHEMICAL SUITS AND ENCLOSURE MATERIALS PHASE II

TRI

ESTIMATING REMAINING LIFE IN BIOLOGICAL/CHEMICAL SUITS AND ENCLOSURE MATERIALS PHASE II

AHPCRC Contract No. DAAH04-93-C-0012 (CLIN 005)
Final Report

October 11, 1995

Prepared for:

Army Research Office
4300 S. Miami Blvd.
Research Triangle Park, NC 27709

Prepared by:

Texas Research Institute Austin, Inc.
9063 Bee Caves Road
Austin, Texas 78733

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
11 Oct 95

3. REPORT TYPE AND DATES COVERED
Final Report 8-16-93 to 8-15-95

4. TITLE AND SUBTITLE
Estimating Remaining Life in Biological Chemical Suits and Enclosure Materials Phase II

5. FUNDING NUMBERS
DAAH04-93-C-0012

6. AUTHOR(S)
Alan V. Bray

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Texas Research Institute Austin, Inc.
9063 Bee Caves Road
Austin, TX 78733

8. PERFORMING ORGANIZATION REPORT NUMBER
N7507:RAR-D183.5

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Army Research Office
4300 S. Miami Blvd.
Research Triangle Park, NC 27709

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARO 30938.1-MS-SB2

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
CRITICAL TECHNOLOGY
~~Limit~~ distribution to U.S. Government Agencies Only
13 FEB 1996

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

The overall goal of this Phase II portion of this SBIR project was the development of a field NDI kit which uses optimized penetrant systems to detect defects in protective clothing materials. An adjunct goal was to investigate the feasibility of using penetrants to determine the extent of decontamination achieved. A classic penetrant approach to NDE of Teflon and similar protective clothing materials was developed. Penetrant development and testing, then proceed to full-suit testing, conducted under laboratory conditions. The types of penetrant(s) to be used and the method of application were investigated. Next kits utilizing these developments were developed which focus on effective field use of the inspection technique. An iterative process was folowed which allowed for optimization. Later demonstrations of a pure NDE technology and one combining a decontamination effectiveness function were demonstrated at Dugway Proving Grounds, and it became clear that the combination had much wider application than either function alone. Demonstrations of the technology to fire departments and Hazmat teams were met with the same reaction - the decontamination verification function was viewed as an area needing innovation. The result of these inputs was the development of Decon-Check TM, a protective suit decontamination verification and NDE system.

14. SUBJECT TERMS
Nondestructive, Biological, Chemical Suits, Penetrant Decontamination

15. NUMBER OF PAGES
70

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

List of Figures

List of Tables

1.0 EXECUTIVE SUMMARY

This project addressed the development of a fluorescent penetrant-based nondestructive inspection (NDI) technique which can be used in the field to assess levels of damage for chemically protective (CP) clothing and shelters constructed of coated materials. In specific,

1. The overall goal of this Phase II project was the development of a field NDI kit which uses penetrant systems to detect actual or impending defects in protective clothing and shelter materials.
2. An adjunct goal was the investigation of the feasibility of using penetrants to determine the extent of decontamination achieved providing a quantitative basis for improving protective clothing decontamin- ation and techniques.

The outcome of the project resulted in a product that benefits both Army and Hazmat teams worldwide. The project began as an effort to determine the remaining life of chemical and biological warfare (CBW) suits using nondestructive evaluation (NDE) methods, and the final product retains that basic characteristic. In addition the product has also been engineered to provide a positive confirmation of decontamination effectiveness. The need for products that aid in decontamination was reinforced during a visit to the Aberdeen Proving Ground by TRI/Austin personnel. Later demonstrations of a pure NDE technology and one combining NDE with a decontamination effectiveness function were demonstrated at Dugway Proving Grounds. It became clear that the combination had much wider application than either function alone. Demonstrations of the technology to municipal fire departments and Hazmat teams were met with the same reaction as that of Army personnel – the decontamination verification function was viewed as very important and an area needing innovation.

The result of these inputs was the development of **Decon-Check**, a protective suit decontamination verification and NDE system developed by TRI/Austin under Army SBIR Topic A91-127 via Phase I and Phase II contracts. Figure 1-1 is a photograph of the **Decon-Check** Kit which includes the penetrant fluid, the delivery gun and wet scrub brush, carrying bag, and written and video instructions. A total of 25 kits were provided to the Army as part of the Phase II deliverables. **Decon-Check** attributes include:

- **Decon-Check** acts as a disclosing agent – graphically showing the areas which have not been decontaminated after a CBW or Hazmat event.
- **Decon-Check** is applied via compressed air, which can be obtained by SCBA air bottles.
- The **Decon-Check** system uses less water than conventional decontamination schemes by both targeting scrub zones and metering rinse water via a brush applicator.
- Remnants of the penetrant fluid are indicative of compromise of protective integrity – i.e., indicating of cracks, leaking seams, holes, or tears.
- The **Decon-Check** fluid is a powerful cleaning agent – biodegradable solvents, ionic and nonionic surfactants provide broad spectrum detergent activity.

- The thixotropic agent, carboxymethylcellulose, also aids in the re-suspension of contaminants which are washed away in the scrubbing process.
- The viscosity and surface adherence of **Decon-Check** fluid stays in contact with the protective barrier, even with Teflon materials, and acts to improve both disclosing capability and cleaning dwell time.

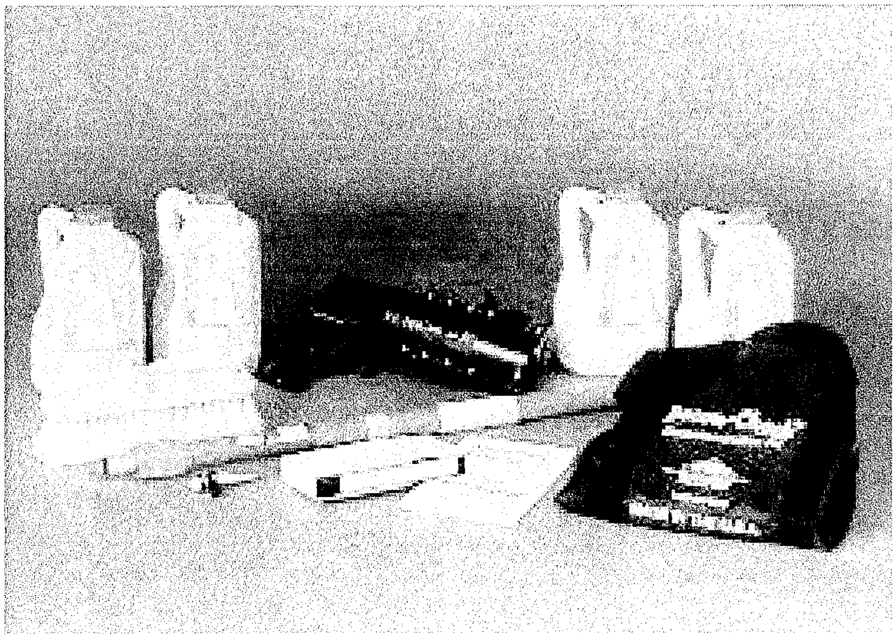


Figure 1-1. Decon-Check Kit

Decon-Check is the first disclosing agent of its type introduced worldwide, and a US patent is pending. In its current form it is a broad spectrum decontamination and cleaning agent, but future development will include agent specific formulations for acid, base, aliphatic hydrocarbon, CBW agent, and isocyanate challenges. **Decon-Check** is available through TRI/Austin and three national safety equipment distributors, sales began July 1, 1995. Sales are expected to be over $1,000,000 by the end 1996. A mailing brochure is included as Figure 1-2, and a formal product brochure may be found in Appendix A.

1.1 Background and Approach

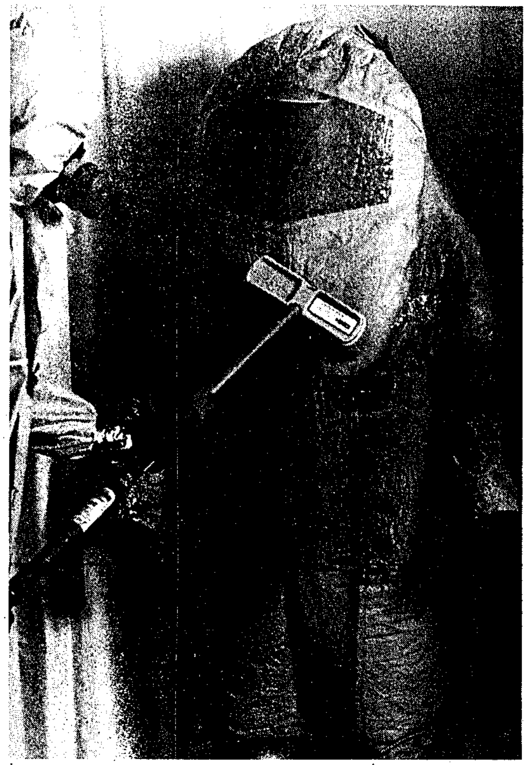
The early stages of the Phase II contract were spent developing a classic penetrant approach to NDE of Teflon and similar protective clothing materials to evaluate barrier integrity. Development strictly along the lines of a penetrant for testing barrier integrity ended at approximately the midpoint of the project. At that time a series of demonstrations to both Army and commercial users of protective clothing were conducted to demonstrate the use and effectiveness of the penetrants. The technological difficulties in applying penetrants to the slippery, non-stick surfaces of protective clothing had been overcome and production planning and product development were just beginning.

Figure 1-2. Decon-Check Mailing Brochure

Visual indication of cleaning effectiveness during decontamination procedures for Haz-Mat protective clothing and equipment.

*Decon-Check is cost effective for incident response and an exceptional **training** aid*

Decon-Check™ shows you where to scrub while its powerful detergent removes surface contaminants.



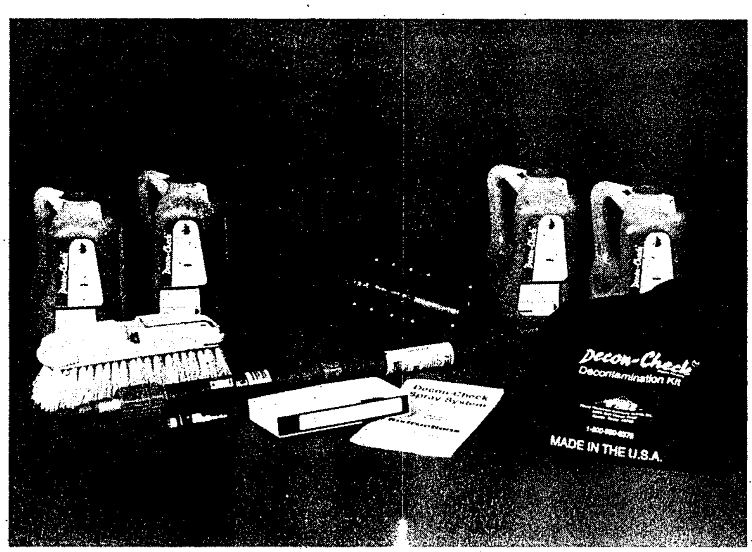
Scrub Contaminants Off

Decon-Check™ Spray System:

- Air powered sprayer uses SCBA or other compressed air source
- Easy "Lock and Load" bottle replacement
- Light, rugged, high efficiency sprayer.

Decon-Check™ Scrub Brush:

- Water-fed brush head
- Adjustable extension handle (2-4 feet) with water control valve



Decon-Check™ Starter Kit

TRI TRI/Austin, Inc.
Decon-Check™ Sales Division

D219.5

Decon-Check™

The first Haz-Mat decontamination detergent/disclosing agent engineered for protective clothing and equipment. The

Bright

Orange

color visually tells you what has been cleaned.

Biodegradable • Non-Toxic • Non-flammable

Call Decon-Check™ Sales Division
TRI/Austin, Inc.
1-800-97-DECON
(1-800-973-3266)
(512)263-2101
Fax (512)263-3530

D219.5

Decon-Check™ Sales Division
TRI/Austin, Inc.
A Texas Research International Company
9063 Bee Caves Road
Austin, TX 78733-6201

At this timeimportance of the Teflon capable penetrant development was found to be secondary to decontamination issues. It was found that suit user exposure to hazardous materials was more likely to occur due to likely to occur due to insufficient decontamination than by fractures, holes, or tears. At Aberdeen Proving Ground Army personnel discussed the problems of both protective clothing and equipment decontamination integrity. This was further supported during Army demon- stration visits to Dugway Proving Ground and Natick Labs, and in a series of meetings and demonstrations with fire departments and municipal Hazmat teams – all users of personal protective ensembles of the similar types to those used in Army CBW. Demonstrations and meetings involving product review of penetrant or **Decon-Check** included:

- US Army Aberdeen Proving Ground, Maryland
- US Army Dugway Proving Ground
- US Army Research Office, Durham, NC
- Fire Fighter Challenge Competition, Austin, Texas
- Houston, Texas Fire Department
- San Antonio, Texas Fire Department
- Austin Texas Fire Department
- Houston Texas Municipal Hazmat Team
- Texas A&M University Fire Academy
- DoD Clothing Research and Development Center, Natick, Mass.
- Air and Waste Management 88th Annual Meeting, San Antonio, Texas
- EPA Region Six Emergency Planning Committee Conference, Amarillo, Texas

To summarize the results of these demonstrations and meetings succinctly is difficult. Some specific quotes are paraphrased below which were helpful in formulation the final product:

- Dr. Bob Reeber, ARO: The Army needs multiple use items to minimize logistic loads. What other functions beyond a protective clothing NDE penetrant can the Phase II product be used for?
- Chief James Ashe, Austin Fire Department: Why do you need the ultraviolet light? I can see the defects without it!
- Millie Warren, Dugway Proving Ground: The penetrant is useful, but is a minor problem compared to the decontamination issue. Can the decontamination fluid be chlorine based to provide a more effective CBW neutralizing agent?
- Houston Hazmat: Time is critical in decontamination, the delivery system should be capable of getting a man out of the suit in less than 3 minutes.

The aggregation of these comments and others collected during the demonstration period made it clear that the technology developed to inspect Teflon and other protective clothing materials was valuable, but not solely as an NDE method for determining suit integrity. At the end of the demonstration series a seminal meeting of the project team was held at TRI/Austin and a new set of project goals was established:

1. Develop a penetrant fluid which provides the following functions:
    - A decontamination verification capability
    - A suit integrity NDE check
    - An aggressive surface cleaning agent capable of removing contaminants

- Capable of sticking to Teflon and similar protective clothing materials in order to support verification and cleaning functions.

2. Develop a delivery system which:
   - Can be applied and removed within a 3 minute time span for a single personal protective suit decontamination.
   - Requires no mixing or complex preparation before use
   - Items which in contact with the suit need to be inexpensive as they will be discarded in the event of actual decontamination events.
   - Minimize water usage and thus waste and logistic problems associated with decontamination.

These goals were all accomplished and **Decon-Check** is now available to both Army and commercial customers. The key to the development was listening carefully to the potential users of the product, without that input the result of this development would have been a very functional and efficient NDE penetrant with very limited practical application in either Army or commercial market sectors.

1.2 Decon-Check Status/Future

Decon-Check sales began officially on July 1, 1995. Toward the end of the project it became clear that the Phase II funds would only be sufficient to supply the documentation CDRLs and TRI/Austin began investing its own funds to finalize production engineering and marketing tasks. Through June 1995 this had amounted to $22,750, and is expected to reach $50,000 before the product becomes self supporting. **Decon-Check** is currently distributed by TRI/Austin and Vallen Safety, and final distribution agreements are being made with Fisher Scientific and RMC Medical. Vallen and Fisher and are large distributors with over $50 million a year in revenues, and the key to moving **Decon-Check** through their distribution systems is to get into their 1996 product catalogs. That process has begun and TRI/Austin anticipates being in at least three 1996 catalogs.

A listing of **Decon-Check** products and suggested retail pricing is included as Table 1-1. The Army can purchase **Decon-Check** through TRI/Austin or its distributors. The initial delivery to the Army includes 25 kits at no additional contract cost. Should the Army elect to establish a Phase III contract with TRI/Austin to provide **Decon-Check** at a reduced rate (as a consequence of large volume buying) the costs listed in Table 1-1 would be reduced to between 5 and 10% off of the retail price.

TRI

8/1/95
Replaces 7/10/95

Table 1-1. Decon-Check Products and Suggested Retail Pricing

Decon-Check™ User Price List

Item	Description	Part Number	Suggested List Price*
1	Decon-Check® Starter Kit	1000	$295.00
2	Decon-Check® Refills (Four 2.2 Liter [2.3 qt] Bottles)	1100	$45.00

Replacement parts:

Item	Description	Part Number	Suggested List Price*
1	Brush	1010	$14.00 per brush head
2	Extension Handle	1020	$27.25 per handle

The **Decon-Check® Starter Kit** contains the following items:

Item	Quantity	Unit	Description
1	4	ea	2.2 liter (2.3 qt) bottle of Decon-Check®
2	1	ea	Brush Head
3	1	ea	Extension Handle
4	1	ea	Air Powered Sprayer & Valve Assembly
5	1	ea	Duffle Bag
6	1	set	Written Instructions
7	1	ea	Instructional Video

- Applicable taxes are not included in the above quoted prices.
- All Prices F.O.B. shipping point.
- All Prices in U. S. Dollars

*- Every effort will be made to keep these prices in effect thru the end of 1996. However, TRI/Austin reserves the right to change these prices upon thirty days notice in the event of unforeseen market changes in material costs.

Product Weights and Dimensions

Item	Description	Part Number	Box Dimensions	Quantity per Box	Weight per box
1	Decon-Check® Starter Kit	1000	7" x 13" x 33 1/2"	1 ea	30 lb
2	Decon-Check® Refills	1100	10" x 11 1/2" x 11 1/2"	Four 2.2 liter (2.3 qt) bottles	22 lb

Replacement Parts:

Item	Description	Part Number	Box Dimensions	Quantity per Box	Weight per box
1	Brush	1010	7 1/8" x 10 3/4" x 13 1/2"	6 ea	7 lb
2	Extension Handle	1020	3 3/8" x 5 3/4" x 30"	6 ea	6 lb

Decon-Check™

Biodegradable • Non-Toxic • Non-flammable

Call toll free **1-800-97DECON** {1-800-973-3266} for ordering or technical information.

Decon-Check™ Sales Division
TRI / Austin
9063 Bee Caves Road
Austin, Texas 78733-6201

1-512-263-2101 TEL
1-512-263-3530 FAX

TRI

Future development in the **Decon-Check** product line will include two primary focus areas:

1. Agent specific versions of **Decon-Check** for military and industrial clients. These developments will be launched when the markets for the agent specific version are assessed as being sufficient for a company funded effort or support for development is provided by the client. In the case of the Army this could take the form of a large sole source order for an agent specific formulation or a sole source development contract. Discussions to date have included:

CHALLENGE AGENT	FORMULATION BASE
Aliphatic Hydrocarbons	(Light) Alcohol
CBW Agents	Chlorine
Isocyanates	Ammonia
Acidic Solutions	Sodium Hydroxide or Other Bases
Basic Solutions	Acetic or Similar Light Acid

2. Advanced high volume delivery systems, capable of decontaminating large numbers of personnel or equipment in a short time period. These systems would have the advantage of high volume personnel/equipment decontamination, low waste generation, and minimal water usage. Potential clients for development include NASA (fueling teams), industries which utilize protective clothing routinely in the production of hazardous chemicals (e.g., the chemical aniline, used in inks, is extremely poisonous and often produced by personnel in full PPE), and the military for CBW applications.

   Both of these approaches will be submitted to the Army under a sole source Phase III proposal. Successful completion of a Phase II project is sufficient sole source justification for award of a Phase III contract. Phase III funding origins are operational in nature, i.e., are not funded from the SBIR pool.

2.0 INTRODUCTION

2.1 Background

Phase I demonstrated the feasibility of using nondestructive inspection (NDI) penetrant-based methods for detecting fatigue cracks and similar breaches of integrity in chemical protective clothing and shelters. This provides a direct means of determining remaining life in CP suits and shelters. CP materials range from treated textiles to highly engineered multi-layer, multi-film composites. A selection of CP materials were tested using off-the-shelf penetrant systems normally used for inspecting metallic surfaces. Microscopy, biopenetration, and chemical permeation were used to verify the results of penetrant NDI testing. CP materials were fatigued using ASTM methods. Controlled diameter and depth punctures were also used to induce material defects.

Phase II provided a further opportunity to develop these unique materials for CP inspection. Formulations were developed using both dye and pigment base

formulations. Fluorosurfactant and other wetting agents were found to provide excellent wettability of PTFE and PE suit materials. The optimized formulations were used for full-suit inspections. Field trials were performed at military and non-military sites.

2.1.1 Problem Statement

Reusable chemical protective clothing suits must be decontaminated and inspected prior to reuse. Improved technologies that enhance the ability of inspectors to determine the location of any defects are critical to the safety of personnel wearing the CP clothing. Reliable techniques for both finding defects and enhancing the ability to decontaminate protective materials have been lacking.

Matching the developed penetrants to a wide range of CP materials was the significant Phase II challenge, but the Phase I results showed clearly that it is a feasible objective.

During Phase I it was noted that during application of the penetrant, the commercial versions tested tended to bead and required continual rebrushing to ensure coverage of the area being inspected. This problem was addressed by developing a higher wetting penetrant system. In addition, a higher wetting penetrant system was found to wick to a greater degree thus enhancing defect detection.

Commercial applications of this technology abound in the protective clothing industry. As an example, in hazardous waste clean up there is a problem with determining the viability of suits which have been used and then cleaned. The methods developed are directly applicable to the commercial protective clothing remaining life problem.

DoD and commercial users of protective clothing have faced the same problem: the integrity testing and certification process ends when the end user receives the CP suit. The Phase II products developed provide these users a means of field testing CP suits and enclosures that reduce replacement costs, ensure suit protective functionality, and save lives.

2.1.2 Project Objectives

As chartered in the Phase II proposal this project addressed the development of a penetrant-based nondestructive inspection (NDI) technique which can be used in the field to assess levels of damage for chemically protective (CP) clothing and shelters constructed of coated materials specifically:

1. The overall goal of the Phase II project was the development of a field NDI kit which uses optimized penetrant systems to detect actual or impending defects in protective clothing and shelter materials.

2. An adjunct goal was to investigate the feasibility of using penetrants to determine the extent of decontamination achieved – providing a basis for improving protective clothing decontamination procedures and techniques.

The primary intent of the project was the development of a product to be used to determine the remaining life chemical and biological warfare (CBW) suits using nondestructive evaluation (NDE) methods. During the course of the Phase II effort the second objective, that the product should provide a positive confirmation of decontamination effectiveness, became the primary goal.

2.2 Approach

Initial work focused on a broad scope of preliminary candidate materials that were chosen for screening evaluation. This initial evaluation was intended to determine the ability of fluorescent penetrant testing on a wide selection of materials. Later in the project formulations that performed a dual function of decontamination and flaw detection were developed. In both cases formulations that were able to wet low energy surfaces such as Teflon and polyethylene were sought.

The approach began with penetrant development and testing, then proceed to full-suit testing, conducted under laboratory conditions. First, the types of penetrant(s) to be used and the method of application were investigated. Next, kits utilizing these developments were developed which focus on effective field use of the inspection technique. An iterative process was followed which allowed for optimizing:

- Penetrant selection;
- Method development; and
- Kit design.

The success in optimization was measured by comparing penetrant/application and technique/kit design against specific goals.

The approach for developing an optimized penetrant was to work with a group of commercially available penetrant systems and to modify their formulations to accommodate the different polymeric surfaces of CP clothing and enclosure materials. This was done by investigating the effects of wetting agents and other surfactants on penetrant formulation and detection. There were two approaches:

1. Direct addition of wetting agents and surfactants to commercial penetrant systems; and
2. Start with the fluorescent material separated from the commercial carrier liquid and develop a new high wetting carrier.

Sherwin, Inc., our Phase II partner, was involved in many steps of Phase II for penetrant development such as a:
- Supplier of raw materials;
- Technical consultant in the admixing of fluorescent suspensions; and
- Source for production engineering information in penetrant development.

Both formulation approaches were required to develop optimized penetrant systems. As in Phase I, penetrant performance was compared with results from permeation testing of material samples. Therefore, this stage also encompassed selection of :

- Key clothing/shelter materials used in both military and commercial applications;
- Conditioning techniques for fatiguing material specimens prior to testing; and
- Methods for ascertaining degradation of material performance indicative of limited service life.

Following the optimization of penetration systems in Stage 1, work proceeded to devising methods for extending the penetrant techniques to whole products. To demonstrate real utility of the penetrant-based inspection technique, complete items of protective clothing were inspected.

After penetrant optimization, work focused on developing prototype inspection kit(s) for both penetrant and **Decon-Check** formulations. The kits were configured such that all components fit within a light weight portable carrying box. First article kits were developed and sent to a limited number of selected test sites including two military installations. TRI/Austin personnel accompanied the kits to the evaluation sites to oversee use.

A total of 25 pre-production kits were also assembled. These kits include improvements based on recommendations from first article kit evaluation. These kits were sent to a larger number of test sites, including a total of 12 facilities within both the private and public sectors.

Commercialization involved two specific areas:

1. Scale up for production of kits; and
2. Marketing to prospective government and commercial clients.

Implementing kit assembly at production levels entailed scale-up of kit contents from small batch quantities to larger quantities to fulfill production quota needs. The remainder of kit production planning involved negotiating price breaks for kit components and specifying assembly/distribution responsibilities.

The second part of commercialization was the development of a marketing plan. This plan identified both government and commercial markets. Target areas included:

- Facilities which acquire and maintain chemical protective clothing;
- Chemical munitions depots;

- Explosive ordinance disposal groups; and
- Government emergency response teams.

The marketing plan also addressed:

- Assessment of market size and potential user groups;
- Expected sales over five year period;
- Kit production quotas needed to meet sales projections;
- Methods for advertising availability;
- Method of distribution; and
- Replacement parts.

3.0 PENETRANT SYSTEMS DEVELOPMENT AND EVALUATION

3.1 Review of Phase I Findings

The most startling finding in Phase I was that ultraviolet (UV) penetrants applied to Teflon®/Nomex® or fiberglass/Teflon® and similar CP composite laminates formed large florescent halos around defects which had breached a layer of Teflon®. These halos show through the translucent coating and are the result of penetrant wicking into the fibrous center layer. The detection probability for defects which breached the outer layer (typically 0.127 mm thick) was virtually 100%. Halo indications were typically 7 mm in diameter for a 0.12 mm puncture defect – an amplification factor of over 50. These results correlated with microscopy tests – when fatigue cracks were seen microscopically the penetrant detected them every time. The same was also true of biopenetration – when a fatigued sample failed biopenetration the same fatigue level sample failed penetrant testing. Penetrant NDI results also correlated well with chemical permeation results. In every instance in which all three replicates in a chemical permeation test showed breakthrough of the challenge substance the penetrant also showed significant defects. This was true even in controlled punctures which were half as deep as the material thickness.

Other CP suit/enclosure material types were also tested, including PVC and butyl coated fabrics. Halos also occurred in these materials and were easily detectable except in materials with high fluorescent backgrounds, such as hot pink PVC. Visible penetrants formed halos in this PVC but left stains.

The Phase I results show clearly that penetrant-based NDI methods detect defects in CP materials at high detection rates. Optimization of these methods to the polymer coatings of CP materials in Phase II resulted in a reliable means of determining remaining life in CP suits and shelters. Phase I preliminary investigations of surfactant modifications to off-the-shelf penetrants showed promise in matching penetrant formulation to polymer surface chemistry for CP material NDI purposes. The Phase I inspections were limited to flat material samples which were tested using a stretching frame. Phase I also made clear the possibility of using penetrant NDI techniques to qualify and QC/QA procedures used in the CP suit decontamination.

3.2 Penetrant System Performance Factors

The first work element entailed a careful review of Phase I findings for identifying those parameters which affect penetrant performance. These consisted of not only the components of the penetrant-based inspection system, but also the physics and chemistry of the penetrant-based detection technology. The second work element required development of a test plan for evaluation of CP material optimized penetrants. Incumbent in this test plan was the selection of materials, conditioning techniques, and conventional material testing techniques against which to judge penetrant performance. Candidate penetrants were prepared by adjusting the formulation chemistry of penetrants shown successful in Phase I and new additional penetrants based on the parameters identified in the first work element. Finally, using the test plan these candidate penetrants were evaluated against performance criteria.

In application of the penetrant, the commercial versions tested tended to bead and required continual re-brushing to ensure coverage of the area being inspected. By developing a higher wetting penetrant system the problems associated with application of the penetrant were eliminated. In addition, a higher wetting penetrant system was found to wick to a greater degree and thus enhanced detection of defects.

Attention was directed towards:

- The effect of penetrants on full clothing/shelter systems;
- Methods for applying penetrant to whole products; and
- The effectiveness of the penetrant system in detecting defects in the complete item.

With respect to clothing/shelter configurations and components, the penetrant-based inspection technique should:

1. Allow evaluation of all types;
2. Not contribute to any product degradation or retention of penetrant in intact materials; and
3. Provide a consistently high level of detection sensitivity and precision for suit or enclosure materials in repeated inspections.

A method was developed which allows the efficient application of penetrant to the entire clothing item. In some inspections, the user may wish to evaluate only a portion of the product. The technique though must also allow inspection of the entire product. Methods were developed and optimized for both purposes. Work in this area, focused on:

- Reducing the time needed to apply the penetrant;
- Ensuring uniform coverage of penetrant; and
- And minimizing the resources required to achieve the application technique.

3.3 Preparation of Candidate Penetrants

3.3.1 General Methodology

During the course of this multi-phase project, a number of commercial penetrant systems were evaluated against selected chemical protective clothing and enclosure materials. In general, these penetrant systems were originally developed and optimized for use in the inspection of metallic surfaces. Metallic surface chemistry, for purposes of penetrant applications, can be characterized as polar and hygroscopic. Polymeric surfaces are just the opposite, having non-polar molecules which resist wetting. The classic example of this is the beading which takes place when water is applied to a waxed car as opposed to the sheeting which occurs when applied to an unwaxed car. As a result, penetrant development for metallic surfaces is antithetic to the development of a penetrant system for polymeric surfaces.

Penetrant detection rates are highest when the penetrant is matched to the inspection surface. The diversity of materials used in chemical protective clothing and shelters makes the job of matching penetrant to material a potentially endless process, and a look at the number and types of penetrant systems on the market is clear evidence of this.

Two methods were used for producing defects in the materials.

1. Using fine gauge (0.12 mm and 0.2 mm) acupuncture needles for creating holes either completely or partially through the material samples; and
2. Reproducibly flexing material samples to varying levels of fatigue per ASTM F 392, "Standard Test Method for Flex Durability of Plastic Films," employing a Gelbo Flex Tester.

Changes in material performance resulting from these defects were ascertained by permeation testing per ASTM F739, "Standard Test Method for resistance of Protective Clothing Materials to Permeation by Liquids and Gases."

3.3.2 Trip to Subcontractor Facilities

TRI/Austin personnel traveled to Sherwin, Inc. in L.A. California on May 14 through May 18th, 1994. The purpose of the trip was to review the subcontractors progress on the development of fluorescent penetrants for use in flaw detection on chemical protective clothing and tentage materials. Other subjects discussed with Sherwin personnel included pricing of penetrants, and development of fluorescent decontamination indicators.

3.3.3 Initial Meeting

TRI/Austin and Sherwin personnel discussed the procedures being used for the evaluation of penetrants. Sherwin indicated that an existing emulsifier product, KO-19, may be used for removal of penetrant after dwell. The use of a brush for removal of penetrant was also advised.

Rejection/acceptance criteria were also discussed. The consensus was that the only viable criterion for determination of a positive defect is the presence of a halo. Other indications such as "headlights" are unreliable because of background noise.

Problem areas on full-suits were discussed. These areas include:

- Seams – because the heat-seaming process results in a solid mass that leaves the material impenetrable to liquids. This eliminates halos.
- Velcro closures – removal of penetrant from these may be a problem.
- Application of penetrant under the cuff section on the suit legs.

All concurred that the inspection techniques, in addition to the pressure test, currently used on full-suits, needed clarification. In addition, how defects are located after failure using the pressure test was discussed in detail.

3.3.4 Laboratory Trials at Sherwin Inc.

Sherwin personnel were given flat sheet stock samples of Challenge 5000 and Challenge 5800/6400 for inspection. TRI-C (dye based) penetrant was used for these inspections. These specimens had 0.004 inch through-hole defects placed at known locations. After application of penetrant and inspection the specimens were tentatively graded using the key overlays. False positives were minimal and true positives were above 50%.

Teflon laminate seam specimens with through holes were inspected with TRI-C with no indication of halos. Saranex samples were also inspected using TRI-A (pigment based). K019 cleaner/remover was used to successfully remove pigment particles left after penetrant application. TRI-A was used for inspection of NASA and CPE materials. No staining was observed after water washing. Although "headlight" type defects were observed, indications of overwashing were observed. Cleaner/emulsifier KO-19 appears to improve washability of TRI-C from the Teflon laminates.

3.3.5 Preliminary Full-Suit Inspection

A preliminary Challenge 5800/6400 full-suit inspection was also performed while visiting Sherwin, Inc.. After pressurizing the suit to 3 inches water the following procedure was performed on the right leg of the suit:

1. Cleaned with Sherwin DR-61. Let dry 5 minutes after wiping.
2. Applied TRI-C to a 6 inch x 6 inch area of the right leg. Washed with water after 10 minute dwell. Inspection revealed significant background coming from residual penetrant imbedded in wrinkles.
3. Re-pressurized the suit to 3 inches water. The area was thoroughly cleaned using water, KO-19 and a brush. A 0.004 inch through hole was placed in the same area. A positive soap-bubble test was observed at the defect location. After three minutes the pressure was 2.95 inches water.
4. Cleaned area again with DR-61 followed by application of TRI-C penetrant and 10 minute dwell time.

The 0.004 inch through hole was placed in the suit while inflated, after which TRI-C penetrant was applied, allowed to dwell for 10 minutes, and washed off with water. A small "headlight" was observed at the location of the defect. No halo was observed on the exterior of the suit. Inspection of the interior of the suit did show a halo at the defect site.

A significant outcome of this test was that the suit passed the pressure test recommended by the manufacturer with the 0.004 inch hole. The initial pressure was 3 inches water and final pressure was 2.95 inches water. The minimum allowable pressure after 3 minutes is 1.6 inches water.

3.3.6 Chemical Protective Clothing Materials

Twelve preliminary candidate materials were chosen for screening evaluation. This initial evaluation was intended to determine the ability of fluorescent penetrant testing on a wider selection of materials. In order to accomplish this, three primary tests were used, contact angle, fine gauge puncture, and razor cut. The full list of the twelve materials is shown in Tables 3-1 and 3-2.

The materials have individual characteristics which create different penetrant inspection problems, including over-washing, staining, and wettability. With the exception of the Teflon materials, the test fabrics are not completely impermeable to penetrants which results in surface staining. In some cases the fluorescent background left on the surface is sufficient to deter reliable reading of defects.

Razor-cut flaws in the rubber based materials result in shallow defects and resealing of the defects. Since the penetrant entrapment is shallow, it is more susceptible to removal from the flaw during the wash step. The Teflon materials while not subject to staining were found to be much more difficult to wet.

Table 3-1. List of Original CP Clothing Phase II Material Candidates and Their Characteristics

Material	Composition	Color/Translucent	Thickness (mil)	Applications	Source	Point of Contact
Challenge 5000	Multilayer Teflon laminate on both sides of woven Nomex	Orange/white (1)	20	Liquid splash protective suits (military/commercial)	ChenFab, Inc. Merrimac, NH	Mark Sinofsky (603) 424-9000
Challenge 5200	Multilayer Teflon laminate on both sides of woven Nomex	Blue/buff (2)	16	Vapor-protective suits (no longer in use); Will serve as project control	ChemFab, Inc. Merrimack, NH	Mark Sinofsky (603) 424-9000
Challenge 5800/6400	Multilayer Teflon laminate combined with two woven fiberglass substrates	Orange/buff (1)	18	Vapor-protective suits (STEPO/commercial)	ChemFab, Inc. Merrimack, NH	Mark Sinofsky (603) 424-9000
CPE	FR treated chlorinated polyethylene on both side of polyester	Orange/orange (0)	22	Liquid splash-protective suits (commercial)	Standard Safety Equipment Co. Palatine, IL	Keith Nelson (708) 359-1400
MIL C 12189	Butyl rubber coated Nylon twill	Olive/olive (0)	14	Toxicological agent protective aprons, coveralls, hoods, suits, and shelters (military)	Alan Rubber Company Philadelphia, PA	Dan Silvestri (215) 739-6500
MIL C 38149C	Chlorobutyl coated Nomex	Gray/white (1)	21	Chemical protective suits (NASA/Air Force PHE)	Fairprene Fairfield, CT	Michele Tomaino (203) 259-3351

Table 3-2. List of Original CP Clothing Phase II Material Candidates and Their Characteristics (Continued)

Material	Composition	Color/ Translucent Layers	Thickness (mil)	Applications	Source	Point of Contact
PVC	Polyvinyl chloride coated Nylon (one side)	Green (0)	15	Liquid splash-protective suits (commercial)	Safe-Pro Cleveland, OH	Sal Geraci (216) 941-3959 Ext. 3165
Responder *Disposable Material	Polyethylene laminate on both sides of nonwoven polypropylene	Blue/white (1)	20	Liquid splash and vapor-protective clothing (commercial	Life Guard Guntersville, AL	Phil Mann (205) 582-2119
Trellchem VPS	Neoprene/butyl rubber coated Nylon with plastic laminate	Yellow/ yellow (0)	16	Vapor-protective suit (commercial)	Trellborg, Inc. Cleveland, OH	John Schramko (216) 963-0310
X21	Multilayer Teflon laminate on both sises of Kevlar substrate	Green/buff (1)	14	Chemical warfare protective shelters	John Romanowski & Assoc. Inc. Londonderry, NH	John Romanows-ki (603)432-4411
Saranex *Disposable Material	Woven HDPE(SCRIM) coated with LDPE and SARANEX Film laminated in between	Natural/ clear (2)	12	Chemical warfare protective shelters	John Romanowski & Assoc. Inc. Londonderry, NH	John Romanows-ki (603) 432-4411

3.3.7 Penetrant Formulations

Five types of fluorescent penetrants were developed during this phase of the project:

1. TRI-A – Fluorescent pigment suspended in a combination of nonionic surfactants plus 1 to 2% fluorochemical surfactant. This formulation is readily water-soluble.
2. TRI-B – Same as "TRI-A" but less water soluble. The product was formulated for manual wipe-off using a dry cloth or toweling, followed by wiping with a

water saturated cloth. Use of this penetrant is more suited to areas subject to wear and excessive flexing, such as elbows and knees, because of manual processing.

3. TRI-C – Fluorescent dyestuffs dissolved in a combination of nonionic surfactants plus 1-2% fluorochemical surfactant. This product is appropriate only for the evaluation of the Teflon materials, as it stains the other candidate materials. The control of the water wash step is not as critical when using this penetrant.
4. TRI-D – Commercial Sherwin SG-6 with 1% fluorochemical surfactant.
5. TRI-E – This penetrant is a combination candidate containing both fluorescent pigment and dye with fluorosurfactants. The goal with this formulation was to obtain the "halo" defect ability and simultaneously enhance the ability to detect the surface defects with fluorescent pigments.

UV/visible spectroscopic analysis of the fluorescent yellow dye that is used in the penetrant formulations was performed. The resulting scan is shown in Figure 3-1.

3.3.8 Contact Angle Measurements

Work was performed to evaluate the surface wetting properties of the penetrants. The contact angle formed at the intersection of a drop of liquid and, a solid surface can be utilized as a relative measure of a liquids ability to wet that surface. High surface tension liquids tend to exhibit large contact angles, while low surface tension liquids exhibit smaller contact angles. Given the low surface energy materials utilized in protective clothing, it is desirable to minimize the surface tension of any liquid which is to be used as a penetrant. This is necessary to ensure complete wetting of the test surface and proper penetration of all defects.

A technique was developed which enables contact angle measurements of prototype penetrants on the chosen battery of protective clothing materials. These measurements are then compared to standard reference materials and liquids, if desired, and relative comparisons made between penetrants. The technique utilizes a stereo microscope in which a protractor reticle has been installed in the eyepiece. Since the drop of test liquid must be applied to a horizontal surface, a stand was fabricated which allows the microscope head to be fixtured in a position which provides a horizontal view of the drop/surface interface. In addition, a small cylindrical platform was designed which allows spatial adjustment of a sample frame within the field of view of the microscope, thereby allowing angular measurement of the drop/surface interface. Next a technique was devised for mounting a two inch diameter sample of protective clothing material. The circular sample is captured between two flat aluminum rings which are fastened together with thumb screws. To ensure against slippage of the sample between the two rings, one ring is fitted with sharpened pins around the periphery. These pins puncture the sample and are captured in corresponding holes in the other ring. When mounted in this fashion, the sample membrane is further tightened to provide a flat test surface by screwing the sample holder onto the cylindrical stage on top of which is located a one half inch button which exerts a force on the captured membrane.

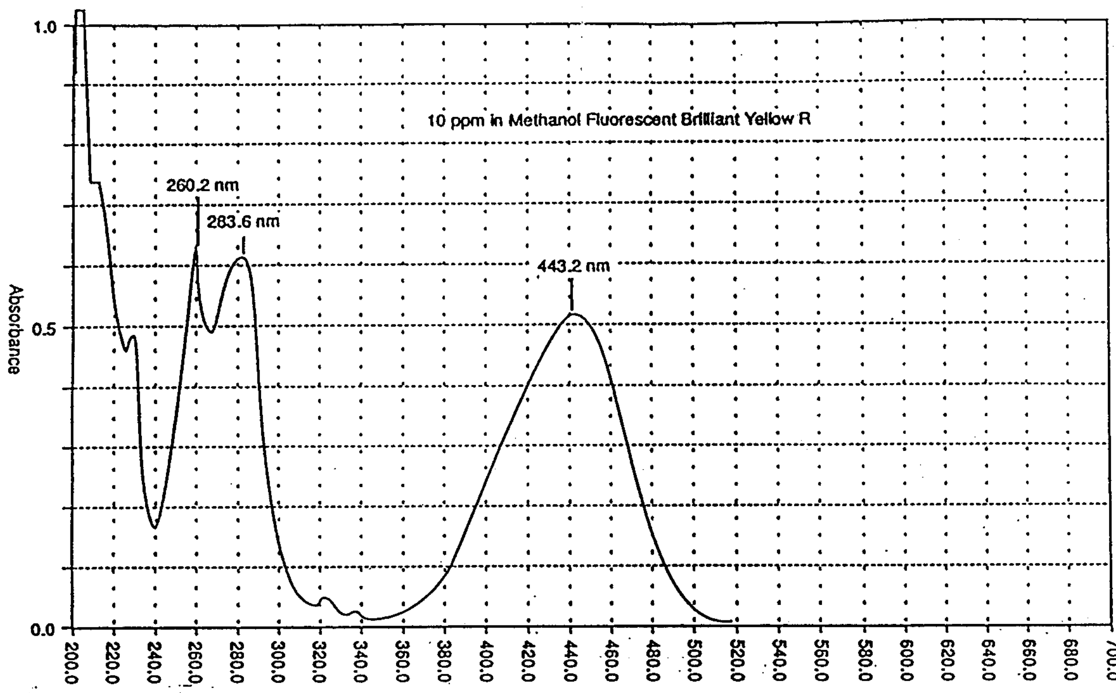


Figure 3-1. UV Absorption of Fluorescent Compound Used in Penetrant Formulations

The contact angle was measured by the following method: the sample is mounted in the ringed sample holder. The sample holder is screwed onto the cylindrical stage, thereby stretching the sample membrane taught. The sample is next cleaned with isopropanol and a clean wiper. After the sample is dry, thirty microliters of the test liquid is pipetted on the stretched, horizontal portion of the sample membrane. The drop/surface interface is brought into focus through the use of the racking mechanism on the microscope head. The sample is next rotated to align the drop/surface interface with the cross hairs of the microscope reticle. At this point the reticle is rotated to determine the contact angle, and the value is read directly from the circumference of the reticle. Given depth of field limitations associated with the microscope, contact angles of less than five degrees may not be determined accurately.

Wettability of each of the twelve candidate materials was determined using the contact angle measuring instrument fabricated by TRI/Austin. Two commercial fluorescent penetrants, SG-6 and ZL-56, and two of the custom formulations, TRI-A and TRI-C, were used for the evaluation. In addition, contact angles were determined using water and isopropyl alcohol. These data may be partially compared to data supplied to TRI/Austin by Chemfab Corporation as shown in Table 3-3. A very good correlation can be seen between the two sets of data giving us confidence that our technique and apparatus are trustworthy.

Table 3-3. Comparison of TRI/Austin Contact Angle Results with Chemfab Corp. Data

Material	Contact Angle With Water	
	TRI-Austin Data	Chemfab Corp. Data
X21	93°	98°
Challenge 5000	106°	95°
Challenge 5200	98°	98°

Table 3-4 illustrates the contact angle results for all materials with water, isopropanol, SG-6, ZL-56, and TRI-A penetrants. This table also indicates whether or not a staining problem was encountered with a particular material. With most of the materials the TRI-A penetrant outperformed the commercial penetrants in terms of both improved wettability and non-staining performance.

Table 3-5 shows similar data obtained on the four primary materials of interest in this study with penetrant TRI-C. The other materials from the "long list" were eliminated from consideration for the reasons shown in Table 3-6. Note that TRI-A penetrant which is pigment based did not generally stain the CP materials, while the dye based commercial penetrants did stain.

Table 3-7 describes the contact angle results obtained using TRI-E penetrant on the Challenge 5000, Challenge 5800/6400, Responder, and X-21 protective clothing materials. Staining was observed only with the Responder material which also exhibited the lowest contact angle. Although the Challenge 5800/6400 did not stain it was relatively difficult to completely remove the penetrant after dwell.

3.4 Inspection Method Development

3.4.1 Sample Conditioning and Permeation Testing

Samples of protective clothing materials from the modified battery were exposed to simulated wear conditions on a Gelbo Flexometer. Three, two inch diameter samples were cut from high stress areas of the aged sample using a die. These samples were evaluated for permeation rate, and time to breakthrough, with a selected permeant. Upon completion of this testing, these samples were reevaluated using penetrant testing to correlate the existence of defects to the permeation rate and break through time. Table 3-8 shows the conditioning levels used and the results of flex conditioning.

Additional samples were prepared from virgin specimens of the modified battery of protective clothing materials. Three, two inch diameter samples were prepared in which a controlled number of half thickness punctures, or punctures of the outer skin were placed. These punctures were induced with a short piece of 0.004 inch acupuncture needle mounted on the tip of a micrometer depth gauge. The utilization of this device allows for very controlled, and accurate application of the punctures. Permeation testing was carried out on these samples in an exact fashion to the previously mentioned samples. Upon completion of the permeation testing, penetrant testing was performed to correlate the rate and break through time to the existence of observed defects.

Table 3-4. Contact Angle Results for All Materials with Water, Isopropanol, SG-6, ZL-56, and TRI-A

Material	Water		Isopropanol		SG-6		ZL-56		TRI-A	
	Contact Angle	Stain Observed?	Contact Angle	Stain Observed?	Contact Angle	Stain Observed?	Contact Angle	Stain Observed?	Contact Angle	Stain Observed?
Butyl/Nylon	82°	No	<5°	No	11°	Yes	9°	Yes	9°	No
Challenge 5000	106°	No	19°	No	47°	No	44°	No	32°	No
Challenge 5200	98°	No	19°	No	48°	No	45°	No	29°	No
Challenge 5800/6400	102°	No	22°	No	48°	No	42°	No	33°	No
Chlorobutyl/ Nomex	72°	No	<5°	No	13°	Yes	14°	Yes	9°	No
CPE/Polyester	78°	No	<5°	No	43°	Yes	28°	Yes	13°	No
PVC/Nylon	75°	No	<5°	No	10°	Yes	8°	Yes	11°	No
Responder	78°	No	<5°	No	13°	Yes	7°	Yes	11°	No
Saranex Laminate (Olive) Front	75°	No	<5°	No	4°	Yes	6°	Yes	<5°	No
Saranex Laminate (Olive) Back	78°	No	<5°	No	15°	Yes	<5°	Yes	<5°	No
Saranex Laminate (Clear) Front	77°	No	<5°	No	5°	Yes	<5°	Yes	<5°	No
Saranex Laminate (Clear) Back	81°	No	<5°	No	12°	Yes	>5°	Yes	<5°	No
Trellchem VPS	111°	No	<5°	No	41°	Yes	27°	Yes	11°	No
X21	93°	No	22°	No	45°	No	48°	No	35°	No

Table 3-5. Contact Angle Results for Five Primary Materials with TRI-C Penetrant

TRI-C Penetrant				
Material	Insp. Side	Contact Angle	Average	Stain Observed?
Challenge 5000	Orange Side	32°,34°,35°	34°	No
Challenge 5800/6400	Grey Side	35°,32°,34°	34°	Slight. UV only. (Wash problem).
Responder	Blue Side	<5°,<5°,<5°	<5°	Yes. UV only.
X21	Green Side	42°,40°,38°	40°	No.

Table 3-6. Materials Removed from Primary Evaluation After Screening Tests

Material	Reason for minimal investigation subsequent to screening tests.
Challenge 5200	No longer in use, used only as a control and a tie-in to Phase I.
CPE	Self sealing nature of the material results in penetrant not reaching the porous inner core of the laminate where wicking eventually results in detectable halos
MIL-C-12189	Same as CPE
MIL-C-38149C	Same as CPE
PVC	Same as CPE
VPS	Staining problems, self sealing, and background fluorescence of the material itself hinders ability to detect halos and flaws.
Saranex (olive)	This is a thin disposable, monolithic material which does not lend itself to detection with the penetrant systems under development.
Saranex (clear)	Same as Saranex (olive)

Table 3-7. Contact Angle Results with TRI-E Penetrant

TRI-E			
Material	Contact Angle	Average	Stain Observed?
Challenge 5000	36°, 37°, 33°	35°	No
Challenge 5800/6400	37°, 41°, 41°	40°	No (Hard to clean)
Responder	9°, 10°, 13°	11°	Yes
X21	45°, 46°, 47°	46°	No

Table 3-8. Results of Initial Material Conditioning

Material	No. Flex Cycles	Observed Damage
Butyl Coated nylon*	N/A**	Minimal (may require abrasion)
Challenge 5000	1,000	Severe creases
Challenge 5200	500	Severe creases
Challenge 5800/6400*	5,000	Moderate creases and blisters
Chlorobutyl coated Nomex*	N/A	Minimal (may require some abrasion)
CPE/Polyester	10,000	Moderate creases; some material loss
PVC coated nylon	10,000	Light creases
Responder	2,000	Moderate abrasion; surface abrasion
Saran laminate (olive)	500	Pinholes created (Halve flexing)
Trellchem VPS*	2,500	Pitting of elastomer
X21*	250	Moderate creasing and blisters

* Key study material;

** Repeated flexing unlikely to create material damage; abrasion/partial puncture offer better means for stimulating material wear

Preparation of the samples was conducted in a way to ensure that the personnel performing the permeation testing and the penetrant testing did not have access to the sample preparation techniques or each others test results prior to performing their respective testing. In this way, an unbiased correlation may be obtained in which the penetrant tests may be evaluated.

Tables 3-9 through 3-11 show the results of the puncture/penetrant tests with TRI-A, TRI-C, SG-6, and ZL-56 penetrants. Baseline seam samples from the five primary study materials have also been evaluated with TRI-A and TRI-C penetrants. These results are shown in Table 3-12. Permeation tests were performed per ASTM F 739. The samples were exposed to the challenge chemicals for four hours at ambient temperature. The breakthrough times reported in Table 3-13 indicate actual system sensitivity.

3.4.2 Evaluation of Seam Materials

Seam material for NASA MIL-C-381-49C, CPE, Challenge 5000, Challenge 5800/6400 was obtained. These materials were fatigue stressed using a Gelbo tester. After fatiguing, penetrant testing was performed as presented in Table 3-14.

3.4.3 Full-Suit Inspection – Responder

Because of the tendency of the Responder material to stain with dye based penetrants, the pigment based TRI-A penetrant was evaluated. A spray bottle was used to apply the fluorescent penetrant. It took 12 minutes to apply penetrant and the total amount used was 32 fluid ounces. After a 10 minute dwell time, an entire container of Sherwin KO-19 foam cleaner was applied and allowed to dwell for 10 minutes. The cleaner was then rinsed from the suit with a water hose with nozzle attachment. The rinsing procedure took 5 minutes.

Table 3-9. Puncture/Penetrant Results for TRI-A Penetrant

		TRI-A				Material Thickness	Hole Depth	No. of Holes
Material Sample	No	Halo Y/N / Diam.	No. of Holes Detected	Dwell Time	Remarks			
MIL-C-12189	1	N	0	5 min.	Overwash	0.011	0.007	4
	2	N	0	5 min	Overwash	0.011	0.007	3
Challenge	1	N	1	5 min.		0.018	0.012	3
5000	2	Y/10 cm, 12 cm	2	5 min.	0.018	0.012		4
Challenge	1	N	4 - 3 large, 1 small.	5 min.		0.010	0.007	4
5200	2	N	3	5 min.		0.010	0.007	3
Challenge	1	N	3	5 min.	Background	0.0200	.015	3
5800/6400	2	N	2	5 min.	Background	0.020	0.015	3
MIL-C-	1	N	2	5 min.	Stain	0.017	0.013	4
38149C	2	N	1?	5 min.	Stain	0.017	0.013	3
CPE	1	N	2	5 min.		0.025	0.020	4
	2	N	0	5 min.		0.025	0.020	4
PVC/Nylon	1	N	0 Stain	5 min	Overwash	0.017	0.013	4
	2	N	0 Stain	5 min.	Overwash	0.017	0.013	4
Responder	1	Void	Void	5 min.	Edgewick	0.016	0.012	4
	2	N	3	5 min.	Edgewick	0.016	0.012	3
Saranex	1	N	2??	5 min.	Hard to see	0.012	0.008	3
(Olive)	2	N	3??	5 min.	Hard to see	0.012	0.008	3
Saranex	1	N	0	5 min.	Overwash	0.013	0.008	3
(Clear)	2	N	0	5 min.	Overwash	0.013	0.008	3
Trellchem	1	N	0 Edgewicking	5 min.	Overwash	0.014	0.010	3
VPS	2	N	0 Edgewicking	5 min.	Overwash	0.014	0.010	3
X21	1	N	3	5 min.	Hard to see	0.010	0.007	3
	2	N	0	5 min.	Hard to see	0.010	0.007	4

Table 3-10. Puncture/Penetrant Results for TRI-C Penetrant

		TRI-C				Material Thickness	Hole Depth	No. of Holes
Material Sample	No	Halo Y/N/ Diam.	No. of Holes Detected	Dwell Time	Remarks			
MIL-C-12189	3 4	N N	2 ?	5 min. 5 Min.	Background, Stain UV only	0.011 0.011	0.007. 0.007	5 3
Challenge 5000	3 4	Y/30 cm, Y/17cm 14 cm, 9 cm	2 (1 thru) 3	5 min 5 min.		0.018 0.018	0.012 0.012	5 3
Challenge 5200	3 4	Y/2cm, 3 cm Y/3 cm, 3 cm,3cm	2 3	5 min. 5 min.		0.010 0.010	0.007 0.007	3 5
Challenge 5800/6400	3 4	N N	3 1	5 min. 5 min.		0.020 0.020	0.015 0.015	4 3
MIL-C-38149C	3 4	N N	2 2	5 min. 5 min.	Background, Stain UV Stain	0.017 0.017	0.013 0.013	5 3
CPE	3 4	N N	0	5 min. 5 min.	Background, Stain UV & V Satin	0.025 0.025	0.020 0.020	4 4
PVC/Nylon	3 4	N N	0 0	5 min. 5 min.	Background, Stain, UV & V Stain	0.017 0.017	0.013 0.013	4 4
Responder	3 4	N N	0 0	5 min.	Background, Stain UV only Stain	0.016 0.016	0.012 0.012	3 4
Saranex (Olive)	3 4	N N	2? Very slight 2 ? Very slight	5 min. 5 min.	Overwash Overwash	0.012 0.012	0.008 0.008	3 3
Saranex (Clear)	3 4	N N	0 0	5 min. 5 min.	Overwash Overwash	0.013 0.013	0.008 0.008	3 3
Trellchem	3	N	2?	5 min.	Background,	0.014	0.010	3
VPS	4	N	0	5 min.	Stain UV only	0.014	0.010	3
X21	3 4	Y/8,10cm N	2 0	5 min. 5 min.	Backside Halo	0.010 0.010	0.007 0.007	4 3

Table 3-11. Puncture/Penetrant Results for Challenge 5000 with SG-6 and ZL-56 Penetrants

	SG-6						Material Thickness	Hole Depth	No. of Holes
Material	Sample No.	Halo Y/N	No. of Holes Detected	Dwell Time	Halo Dia.	Remarks			
Challenge 5000	1 2	Y Y	4 7	1 min. 1 min.	2,3 cm 2,5,4 cm.	2,15 cm Large (Edge) (4 together) 15 cm.	0.018 0.018	0.012 0.012	3 3

	ZL-56						Material Thickness	Hole Depth	No. of Holes
Material	Sample No.	Halo Y/N	No. of Holes Detected	Dwell Time	Halo Dia.	Remarks			
Challenge 5000	1 2	Y Y	3 3	1 min. 1 min.	5,3,2 cm. 5,8,7 cm.		0.018 0.018	0.012 0.012	3 3

Table 3-12. Baseline Seam Evaluation with TRI-A and TRI-C Penetrants

Material Sample	No/Insp. Side	Halo Y/N/ Diam.	No. of Holes Detected	Dwell Time	Remarks Location	No./Insp. Side	Halo Y/N/Diam.	No. of Holes Detected	Dwell Time	Remarks Location
Challenge 5000	1 O 2 W	No N/A	N/A N/A	10 min. 10 min.		3 O 4 W	No N/A	N/A N/A	10 min. 10 min.	N/A N/A
Challenge 5800/6400	1 G 2 W	No N/A	N/A N/A	10 min. 10 min. N/A		3 G 4 W	No N/A	N/A N/A	10 min. 10 min.	N/A N/A
Responder	1 2	N/A N/A	N/A N/A	N/A N/A	N/A	3 4	N/A N/A	N/A N/A	N/A N/A	N/A N/A
X21	1 2	N/A N/A	N/A N/A	N/A N/A	N/A N/A	3 4	N/A N/A	N/A N/A	N/A N/A	N/A N/A

Table 3-13. Permeation Test Results

CP Material	Challenge Chemical	Average Breakthrough Time (min)		
		Pristine	Partial Puncture	Flexed
Challenge 5000	Dichloromethane	78	16	52
Challenge 5200	Dichloromethane	96	12	12
Challenge 5800	Diethyl Ether	64	10	15
X-21	Acetone	32	<4	<4

Table 3-14. Penetrant Test Results for Gelbo Flexed Seam Samples

Material	Number of Cycles	Penetrant Used	Test Results
Challenge 5000	1,000	TRI-C	No Defects Detected
Challenge 5800/6400	5,000	TRI-C	Positive indications of defects
CPE	10,000	TRI-A	Slight indication of defects
NASA MIL-C-381-49C	2,500	TRI-A	No Defects Detected

On inspection of the exterior of the suit it was noted that penetrant had wicked into the interlayer of the material by traveling through the stitches where Velcro is attached at the rear closure area. This resulted in several 2 inch halo patterns.

A problem was also noted with the face shield. The shield is composed of two transparent layers. Somehow the penetrant entered the area between these two films and accumulated approximately 10 cc of penetrant. The Velcro closure material itself appeared to absorb some penetrant but this was removable on rinsing with water.

Spot Inspection Results – Challenge 5800/6400

A Challenge 5800/6400 suit was used for spot inspections of the elbows, knees, armpits, crotch, forearm, and foot. This suit had not been previously worn or conditioned. Table 3-15 summarizes the results of these tests, including amounts of penetrants and cleaners used, dwell and application times, and total test time per area.

Table 3-15. Spot Penetrant Inspection by Area

Private Location (Flat)	Pre-Clean Time DR-60 (Min)	Amt. DR-60 Used (Gm)	Dry Time (Min)	PT Appl. Time (Min, Secs.)	Amt. PT Used (Gm)	Dwell Time (Min)	Removal Time Incl. KO-19 Appl. (Min)	Insp. Time (Min)	Amt. KO-19 Used (Gm)	Post Clean Time DR-60 (Min)	Amt. DR-60 Used (Gm)	Amt. H2O Used (Oz.)	Amt. Wipes Used	Total Time Per Insp. Area (Min)
Elbow	1'	5.23	5'	1' 40"	5.51	10'	6'	4'	33.21	1'	9.45	1	15	29'
Knee	1'	3.03	5'	1' 45"	7.54	10'	6'	7'	13.65	1'	5.13	1	16	31'
Armpit	1'	3.94	5'	2' 0"	5.63	10'	9'	3'	32.96	1'	6.2	1	19	31'
Crotch	1.5'	4.04	5'	2' 0"	7.55	10'	9'	4'	30.79	1'	10.26	1	18	32.5'
Forearm	1'	6.46	5'	2' 0"	7.09	10'	8'	5'	34.88	1'	9.72	1	17	32'
Foot	1'	7.40	5'	1' 0"	3.23	10'	5'	4.5'	13.59	1'	3.95	1	14	27.5'

3.4.4 Level of Detection Experiments

Efforts were undertaken to determine the level of detection capabilities of selected penetrants with the battery of chosen barriers. As a first trial, the Chemfab barrier Challenge 5000 was chosen for investigation. This material is a Teflon laminate with a non-woven Nomex interlayer for strength. Past experience has shown that the dye based penetrant TRI-C provides greater amplification of defects, in the form of halos, when utilized on Teflon laminates. This penetrant was chosen for this study.

The experiment was conducted blind with the samples being prepared by personnel not involved in the inspection process. The preparation procedure consisted of the infliction of controlled punctures of the barrier material by acupuncture needles of varying diameter. These punctures were made using portions of these needles mounted on micrometer depth gauges. In this fashion the depth of puncture could be controlled to ensure only puncture of the challenge, or outer membrane of the material. Three sizes of needles were used in this study 0.004, 0.008, and 0.012 inches diameter. In order to gain a statistically significant measure of the level of detection, it was decided to evaluate thirty punctures at each of the previously described sizes. In order to prevent overcrowding of the test sample it was decided to test six samples each being one square foot in area. In doing so, this allowed 15 punctures to be applied per sample. Each sample contained an assortment of puncture sizes with the total of all samples resulting in 30 punctures of each size hole. The placement of the punctures was determined with the use of Mylar templates which were the same size as the samples being punctured. Alignment holes were placed in the template indicating the puncture position. Surrounding the alignment holes were alignment markings which were color coded to indicate the type of puncture made in the barrier material. Thus, the templates (6 total), provided a means for placement of the test punctures, and was utilized as a means of evaluating the ability of the operator to detect defects in the material after testing. As a final preparation step, it was necessary to wax all edges on all samples in order to prevent edge wicking of the penetrant into the sample thereby obscuring detection.

The samples were tested using the dye based penetrant TRI-C with the procedure described in Appendix B. As defects were detected they were circled with a wax marker to locate the defect for later evaluation. Both "halos" on the back side of the sample, and "headlights" on the front of the sample were noted. The templates were next placed over the top of the respective sample to determine the specific level of detection for each type puncture. Table 3-16 details the results of this evaluation.

In conclusion, a 93.3% level of detection was observed for the smallest (0.004") punctures made in the material with the penetrant TRI-C. A 100% level of detection was measured for 0.008" punctures, and 93.3% for 0.012" punctures. A significant number of false positive results was observed on some samples. The cause of these results is unknown at this time, however, this may be related to the ability of a material to retain penetrant within small patterns on the surface of the material. It may be necessary to limit detection of defects with this material to "halo" type indications.

Additional blind tests were performed on various protective clothing materials in order to establish level of detectability with fluorescent penetrants. Table 3-17 shows the results of these tests. Each material had 30 holes each at 0.004 inches, 0.008 inches, and 0.012 inches in diameter.

Table 3-16. Level of Detection Tests Performed on Chemfab 5000

Detect Size	Sample Number					
	1	2	3	4	5	6
.004" Actual	3	5	5	2	10	5
0.004" Detected	3	5	4	1	10	5
0.008" Actual	2	5	5	10	3	5
0.008" Detected	2	5	5	10	3	5
0.012" Actual	10	5	5	3	2	5
0.012" Detected	10	5	5	3	1	4
False Positive	34	11	9	3	2	29
False Negative	0	0	1	1	1	1
Total Detected	49	26	24	18	17	44

Table 3-17. Level of Detectability, Flat Panel Specimens

Private Material	0.004"	0.008"	0.012"	% False Positives
CPE	0	0	37	43
PVC	3	0	0	37
NASA	3	17	50	153
Saranex Clear	63	53	100	30
Saranex Olive	63	63	97	164
X-21	93	83	100	0
Chemfab 5600	50	50	87	17

The NASA and Saranex olive materials point out the problem of excessive false positives when inspecting the opaque materials. These types of materials do not clearly indicate defects by the "halo" effect and the technician must rely on the smaller "headlight" type defect detection criteria which is much more likely to result in false positives. The most successful result was with the X-21 material (Teflon based) which resulted in no false positives and a 93% detection rate at the smallest hole size.

3.5 Penetrant Inspection Kit Design

3.5.1 Kit Concept Development

Several penetrant inspection kit design concepts were considered during this phase of the project. Concepts pertaining to the contents required for various types of kits were considered. These concepts included:

- Whole suit inspection kits;
- Spot test inspection kits;
- Enclosure inspection kits; and,
- Accessory list.

After considerable evaluation the spot inspection kit was chosen as the first article penetrant inspection kit. It was determined that this type of kit provided the most likely format for a usable product in the field. The primary utility of this kit is for finding defects in critical areas of enclosure suits, such as knees, elbows, and face shield areas. Table 3-18 shows the contents of the spot inspection kit.

Table 3-18. Spot Inspection Kit Contents

1. UV Light
2. Battery Pack
3. Battery Pack Access.
4. UV Glasses
5. Funnel
6. Wipes (1 Box)
7. 2 Pair Gloves
8. Apron
9. 1 Qt. PT
10. 1 Qt Pint PT
11. Small Pump Spray Bottle (Red)
12. 1 Can KO-19
13. 1 Large Spray Bottle (Gray)
14. 2 China Markers
15. 1 Can DR-60 Cleaner/Remover

3.6 First Article Production Field Evaluation

Ten first article penetrant inspection kits were prepared for test and validation. These kits allowed evaluation at several military sites in order to finalize training and instructional materials for the kits. Inspections at these test sites also allowed for debugging and verifying prototype kit operability and field inspection integrity.

The penetrant inspection kit was first introduced informally at Aberdeen Proving Grounds during the Symposium on Nuclear, Biological & Chemical Contamination Survivability (NBCCS, June 15, 1994).

The first field trial of the penetrant inspection kit occurred at Dugway Proving Grounds (DPG) Dugway, Utah in January, 1995. A Protective and Safety Equipment Inspector and a Test Director evaluated the products. DPG personnel were given protective clothing samples of Chemfab Challenge 6400, Responder, and Saranex. The samples each had an unknown (to the tester) number of 0.004" through-holes. In some cases the samples had no holes at all. The results for this blind test are shown in Table 3-19.

Table 3-19. Blind Field Test Results

	6400		Responder		Saranex	
Tester	Actual Holes	No. Holes Detected	Actual Holes	No. Holes Detected	Actual Holes	No. Holes Detected
#1	2	2	none	none	1	1
#2	none	none	2	2	3	3

A 100 percent detection rate was observed for blind tests described above. DPG personnel commented that even with the perforated Butyl level B material that was tested detection of defects was accentuated by using the fluorescent penetrant.

The second field trial with the penetrant inspection kit was performed at U.S. Army Soldier Systems Command Natick (Provisional), Natick, MA. in March, 1995. NATICK has been heavily involved in the development and evaluation of the STEPO (Self-Contained Toxic Environment Protective Outfit). They coordinate field evaluations of the STEPO suits that occur at Dugway Proving Grounds. For this reason they were very interested in products that may be used for the evaluation of defects in STEPO suits. Project objectives and a general overview of progress to date was presented to NATICK personnel. NATICK personnel included two US Army-NRDEC Chemical Engineers and one Systems Engineer with General Technical Services, Inc.

The penetrant inspection kit was described to NATICK personnel prior to laboratory testing. It was explained that penetrant inspection kit was developed as a

defect detection aid for inspection of whole-suit products and now is intended as a spot inspection product. The cleaning and drying procedures used with TRI-C penetrant were briefly summarized. The actual evaluations were performed in on-sight laboratories.

An initial demonstration of the penetrant testing technique was performed using Chemfab Challenge 6400, Responder, and Saranex (clear). Each material contained one through-hole (0.004" diameter). TRI-C penetrant was used for the initial evaluation of these materials.

Comments made by Natick personnel during the penetrant evaluation:

1. *The instruction booklet is easy to follow;*
2. *A larger U.V. light might be useful for inspecting larger areas. A timer included with the kit might be useful also;*
3. *Inspection of knee areas on suits is very time consuming because the leg portion must be turned inside-out;*
4. *The technique appeared to work well on the responder suit and appeared to be less effective on the Chemfab (STEPO) suit;*
5. *Actual use seems to be limited to spot-checking and military applications may be limited;*
6. *The penetrant doesn't seem practical for monitoring whole suits, but might be useful for checking patches for effectiveness;*
7. *The kit works, but a potential problem is the contamination of personnel and work areas with fluorescent material that may interfere with tests.*

4.0 PENETRANT /DECONTAMINATION AID SYSTEMS DEVELOPMENT

4.1 System Performance Factors

The unique surface characteristics exhibited by advanced Teflon composite materials prompted the development of penetrants suited to the detection of small punctures in those materials. These penetrants, while found to be well suited to spot inspection of Teflon laminates, exhibit staining with some conventional suit materials, and are cost prohibitive for whole suit inspections. Development efforts were therefore begun on a product which can be utilized as a decontamination aid for chemical exposure suits and equipment. The purpose of this material is to provide a visual means for determining the extent of decontamination, and detect punctures of protective barriers.

Several characteristics were judged as necessary in the development of the decontamination/penetrant product. The product should contain a pigment rather than a dye as a disclosing agent. Utilization of a pigment lessens the occurrence of suit staining. Once applied to a surface (vertical/horizontal), the product should require some mechanical brushing to be removed from the surface, thereby providing a measure of cleansing thoroughness. Removal should not be possible with a simple, low pressure stream of water. Once brushed, the coating should aid in the removal of contaminants from the suit/machine surface and be miscible in water. Finally, the product should provide some measure of detection of suit puncture, or damage.

Since conventional protective suits are composed of materials which have surface energies substantially higher than Teflon composites, it was thought to be possible that a commodity surfactant would be suited to this application. A search was initiated to identify commodity commercial surfactants which would function as a detergent, and a surface wetting agent. A requirement of the surfactant is the ability to be miscible in water. This constraint limited the search to some nonionic surfactants and anionic surfactants. Sulfated ethoxylate alcohol's were extensively evaluated. These surfactants are noted in the literature as being biodegradable.

Several visible/fluorescent pigments were identified which have suitable characteristics for use in this application. These pigments are highly colored in visible light, thereby providing good contrast with the protective suit, or machinery being cleaned. Additionally, these pigments have a robust signature when viewed in ultraviolet light. This characteristic provides suitable sensitivity to aid in puncture detection. These pigments are materials of commerce which have been used in applications requiring low toxicity characteristics.

Early development work centered on a concentrate product which is mixed with water prior to application. While allowing economy of space, and portability, this concept was found to be inconvenient for customers to use. Therefore, a ready to use premixed product was developed. The premixed product can be applied with various air, and electric powered sprayers as well as manually. In order to achieve a rinse resistant consistency which will adhere sufficiently to vertical, and horizontal surfaces it is necessary to employ viscosity builders. Several water soluble/biodegadable polymers were evaluated for this application.

Prototype formulations were prepared and tested for application characteristics. The prototype formulations were found to perform as designed with respect to application, and removal characteristics. Several air, and electric powered sprayers have been identified as suitable devices for the application of this product. The results of the initial sprayer tests are presented in Table 4-1. Initial puncture and stain testing was performed that indicated that this product had some functionality in finding defects.

Table 4-1. Evaluation of Air and Electric Powered Sprayers

	Test 1	Test 2	Test 3
Sprayer	Wagner® Electric Model 404	Wagner® Electric Model 404	Pace Setter® Air Sprayer (80 PSI)
Spray Time (Full Suit Coverage)	1 min. 52 sec	1 min. 40 sec	1 min. 17 sec
Scrub Time	1 min. 30 sec	2 min. 0 sec	2 min. 3 sec
Rinse Time	1 min. 50 sec	1 min. 18 sec	1 min. 29 sec
Total Elapsed Time	5 min. 12 sec	4 min. 58 sec	4 min. 49 sec
Amount of Decon-Penetrant Used	1000 cc	1000 cc	1800 cc
Water Flow In Brush?	No	Yes	Yes
Average Height of Water In Tub After Rinse	1"	1/2"	1 "
Total Water Used (Scrub and Rinse)	28 gallons	14 gallons	28 gallons

orange original formulation premixed by WPZ on Sept. 30, 1994

4.2 Decontaminating Aid/ Penetrant Formulation

4.2.1 Decon-Check Design Data

A central composite design was created to test a variety of formulations for **Decon-Check**. Table 4-2 shows the design with constituents shown as percentages of the total weight.

4.2.2 Scrub Test

One of the tests done with this battery of formulations was the Scrub Test. In this test, 2 mL of each formulation was tested for its ability to remove a grease pencil mark from the Responder CP suit material. The marks were applied with a weighted grease pencil for uniformity. The sample material was attached to a platform and the **Decon-Check** was applied to the grease pencil mark and allowed to set for 30 seconds. Brushes suspended above the sample platforms were then used to brush the **Decon-Check** back and forth across the grease pencil mark. The number of strokes of the brush needed to remove the mark was recorded for each sample tested. As shown in Table 4-3, the results ranged from 21 to 101 passes.

Table 4-2. Decon-Check Experimental Design

Sample	%Limonene	% CMC	%Pigment	%Witcolate	%PEG	%Tergitol
1	15.1	3.8	3.8	3.8	48.9	24.7
2	10.7	10.7	10.7	12.9	40.7	14.2
3	16.8	4.2	16.8	21.0	25.1	16.2
4	10.7	10.7	10.7	12.9	40.7	14.2
5	16.5	16.5	16.5	4,1	28.9	17.5
6	10.4	10.4	0.4	12.5	49.6	16.6
7	11.1	11.1	24.5	13.3	28.9	11.1
8	4.6	4.6	18.5	4.6	60.1	7.6
9	10.7	10.7	10.7	12.9	40.7	14.2
10	15.7	15.7	3.9	3.9	39.4	21.3
11	4.5	4.5	4.5	4.5	72.8	9.1
12	4.7	4.7	18.9	23.6	42.5	5.5
13	4.6	4.6	4.6	23.2	55.8	7.1
14	17.6	17.6	17.6	22.0	13.2	11.9
15	16.8	16.8	4.2	21.0	25.1	16.2
16	21.7	9.8	9.8	11.8	25.6	21.3
17	4.8	19.2	19.2	24.0	28.8	3.9
18	0.5	12.5	12.5	15.0	59.5	0.0
19	11.1	24.5	11.1	13.3	28.9	11.1
20	10.7	10.7	10.7	12.9	40.7	14.2
21	10.4	10.4	10.4	0.4	51.4	17.1
22	15.7	3.9	15.7	3.9	39.4	21.3
23	4.7	18.9	4.7	23.6	42.5	5.5
24	10.7	10.7	10.7	12.9	40.7	14.2
25	11.3	11.3	11.3	31.5	24.8	10.0
26	4.7	18.8	18.8	4.7	47.0	6.1
27	4.6	18.5	4.6	4.6	60.1	7.6
28	10.4	.04	10.4	12.5	49.6	16.6
29	16.0	4.0	4.0	20.0	36.0	20.01
30	10.7	10.7	10.7	12.9	40.7	14.2

Table 4-3. Decon-Check Scrub Test

Sample #	Scrubs to disappearance of mark
1	26
1	21
2	36
2	40
5	28
5	41
6	32
6	35
7	38
7	34
10	23
10	24
14	30
21	25
15	25
15	30
16	23
16	28
18	88
18	101
21	21
21	25
23	56
23	49
25	26
25	30
26	50
26	58
27	51
27	51

A model was derived from this data which showed that limonene and CMC were the important factors in determining how quickly a given formulation could remove the grease pencil mark. Presumably limonene acts as a cleaning agent and the CMC makes the formulation viscous enough to stay in contact with the sample material. The data and the parameters of the model are shown below. Each constituent was tested to see if it was significant in describing the dependent variable "scrubs", but only limonene and CMC were significant. Table 4-4 shows the results of the model as sums of squares.

Coefficients were determined and the equation that describes the model is:

$$\text{\# scrubs} = 125.7341 - 11.397341(\%\ \text{limonene}) + 0.281734(\%\ \text{limonene})^2 + 0.182795(\%\ \text{limonene})(\%\ \text{CMC}) - 2.2426256\ (\%\text{CMC})$$

Table 4-4. Scrub Test Sums of Squares

Source	df	Sum of Squares	Mean Square	F-Value	P-Value
%Limonene	1	3.252844983E3	3.252845E3	9.486781E1	.0001
%Limonene *%Limonene	1	2.686630907E3	2.6866309E3	7.835442E1	.0001
%Limonene *% CMC	1	4.196600989E2	4.19600989E2	1.2239E1	.0018
% CMC	1	3.514725107E2	3.5147251E2	1.025054E1	.0037
Residual	25	8.572046467E2	3.4288186E1		

Dependent: Scrubs

Using this equation, the surface shown in Figure 4-1 can be generated, showing the relationship between limonene, CMC, and the cleaning ability of the **Decon-Check** formulations. The lower portions of the surface would be predicted to clean the grease pencil mark with the fewest scrubs of the brush. The "F" on the surface represents the limonene and CMC percentages in the final formulation. This shows that the final formulation falls in a minimum area for this model: an area of the highest cleaning efficiency.

When the values for the limonene and CMC in the final formulation (12 % and 22% respectively) are input into the equation, we get a predicted value of 29 scrubs. This is near the low end of the range in the test of 21-101.

4.2.3 Slump Test

A slump test was performed to determine how long different formulations would adhere to a vertical surface before running down the surface. Thicknesses of **Decon-Check** ranging from 0.100" to 0.020" were spread onto a sample material (Challenge, grey) which then was held vertically. Each sample was timed to see how many seconds elapsed before the **Decon-Check** ran below its original lower boundary.

The data was not used in considering the final formulation because it appeared that several things besides the formulation were contributing to the test results. One of the factors was the length of the **Decon-Check** streak on the test material. A longer streak would mean that more **Decon-Check** was available and it seemed to be coalescing and slumping faster that the tests with shorter streaks. Due to the way the test was designed, the length of the streak could not be controlled.

Another thing contributing to the test results seemed to be the quality of the Teflon material being used as the test surface. The way the Decon-Ceck beaded on the material seemed important in determining how soon it would slump. If this was indeed a factor, then the results might not have any meaning on a dissimilar surface. For these reasons, this test was not considered in choosing the final formulation.

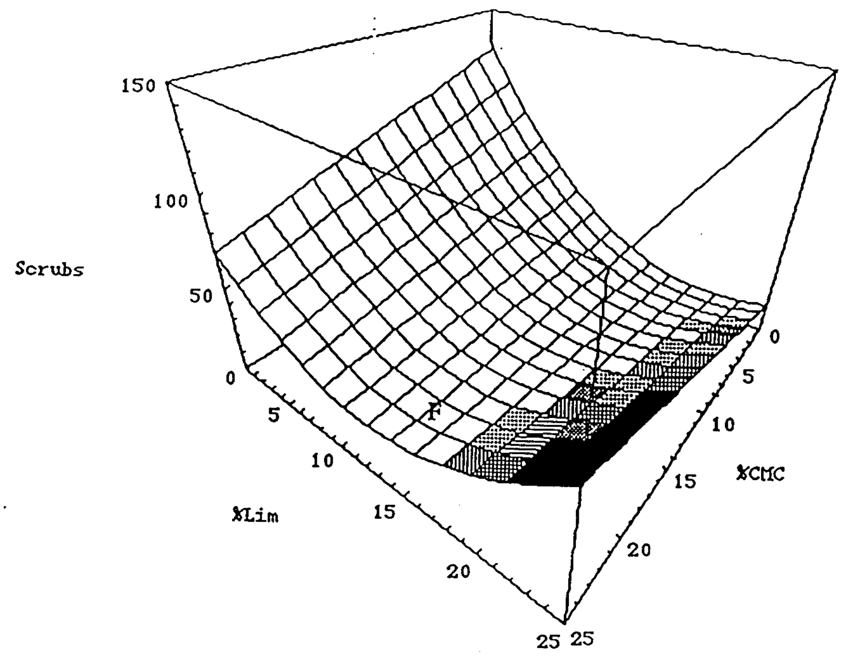


Figure 4-1. Effect of Limonene and CMC on Scrubbing Effectiveness

4.2.4 Viscosity Tests

Viscosity measurements were taken for each formulation in this design on a Brookfield Viscometer with the #3 and #5 spindles. For samples with viscosities above about 100 centipoise, readings were taken after the spindle had been in motion for three minutes. All measurements were made at 22°C. The range of viscosities was approximately 20 to 150,000 centipoise.

A model was derived from the viscosity data, using (ln viscosity) as the dependent variable. The coefficients of the model included terms in limonene, CMC, pigment, and Witcolate. The sums of squares data for these viscosity experiments is shown in Table 4-5.

From the coefficients derived, a model was generated relating viscosity to the relative concentrations of the components in Decon-Check. Figure 4-2 illustrates the excellent fit obtained for predicted and actual viscosity.

4.2.5 Product Stability Study

Multiple preliminary formulations of Decon-Check were evaluated. Performance criteria that were monitored with each iteration included:

- Initial viscosity;
- Temperature dependence of viscosity;
- Degree of settling;
- Performance in pneumatic spray dispenser; and,
- Ease of application and removal from substrate.

Table 4-5. Sums of Squares Data for Viscosity Experiments

Source	df	Sum of Squares	Mean Square	F-Value	P-Value
% Limonene	1	1.409505056	2.542525E1	.0001	
% Limonene * % Limonene	1	1.246806236	1.246806236	2.249042E1	.0001
% CMC *% CMC	1	87.756680096	8.775668E1	1.582992E3	.0001
% CMC * % Witcolate	1	.964043300	.964043300	1.738982E1	.0001
% CMC *% CMS * % C...	1	37.198447686	3.7198448E1	6.710013E2	.0001
Residual	74	4.102854484	.055437223		

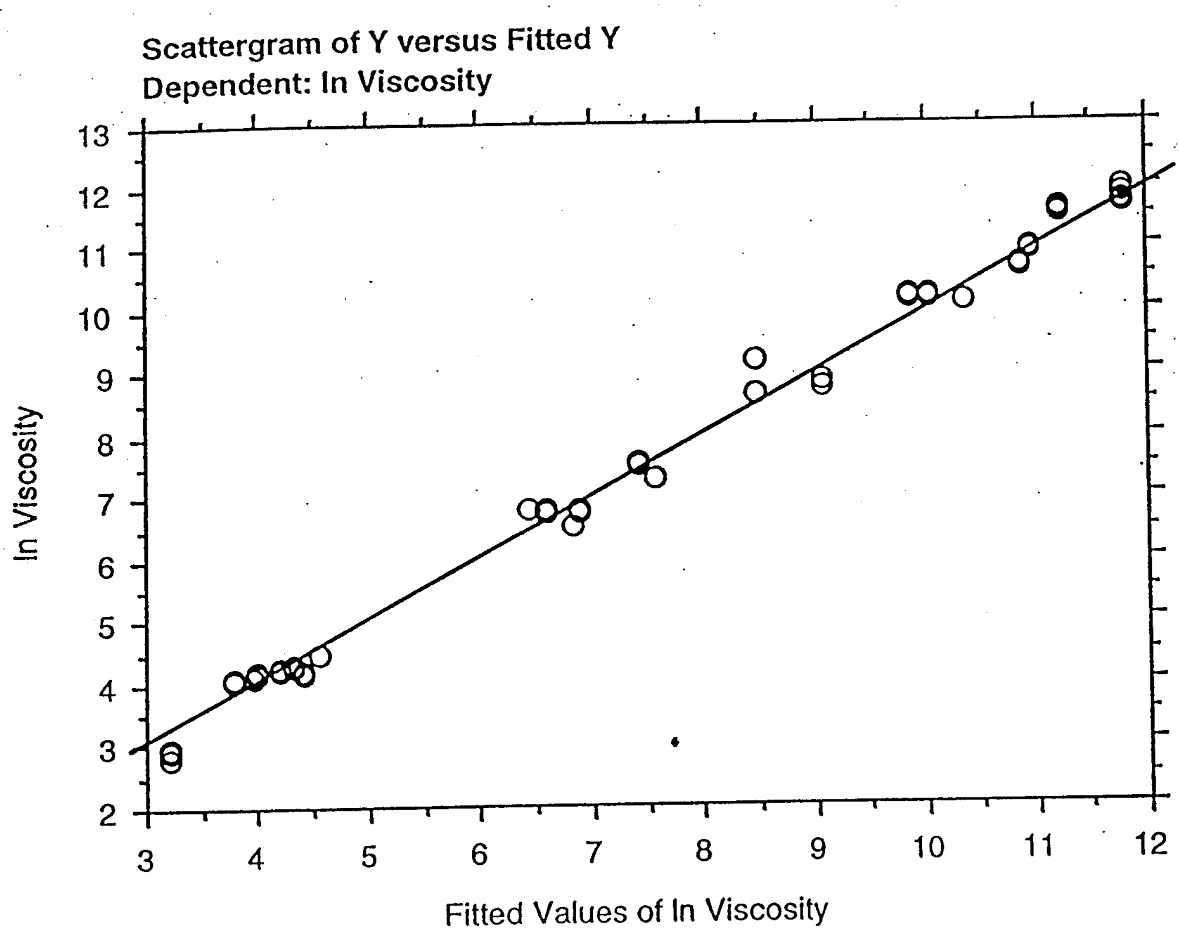


Figure 4-2. Scattergram of Four vs. Fitted Four; Dependent Variable is in Viscosity

After many formulation a satisfactory mixture was found. The concentrated form, that is subsequently mixed with water contains the ingredients shown in Table 4-6.

The composition of Table 4-6 provides an orange-colored concentrate which is mixed with water at an approximate concentration of 1:10 (concentrate: water) to form a suspension.

Table 4-6. Decon-Check Ingredients and the Function of Each

Component	Function
1. Polyethylene Glycol (200 MW)	Solvation of Contaminants
2. Tergitol NP-9	Detergent
3. Witcolate ES-370	Surfactant
4. d-Limonene (Re-distilled)	Solvation of Contaminants
5. Carboxymethyl Cellulose(Sodium) (#7MXF)	Thixotropic Agent
6. T-15 Blaze Orange	Colorant

The resulting suspension is applied to a surface by spraying, such as with a pneumatic or electric sprayer. Alternatively, **Decon-Check** may be applied by manual means. The surface is next brushed to loosen and suspend any surface contaminants present. After brushing, the surface is to be inspected for evidence of incomplete brushing by visually looking for areas where the colored compositions remain undisturbed. Upon complete brushing and inspection of the surface, the surface is rinsed with a liquid, such as a stream of clean water. The surface may then be inspected for evidence of incomplete removal of the coloring agent. This may be accomplished by visually examining the surface under normal white light for the presence of the visually detectable coloring agent. The process may be determined to be complete when no further visible sign of the coloring agent remains. The device or garment that is so processed may then be easily inspected for signs of any surface area that has not been scrubbed as well as for defects in the surface (e.g., for garments seam failure, barrier perforation, etc.) where signs of the coloring agent may be detected.

4.3 Decontaminating Aid/ Penetrant Kit Design

4.3.1 Sprayer Evaluations

After the **Decon-Check** formulation, the sprayer to use for delivery of the **Decon-Check** fluid to the CP clothing surface was perhaps the most critical component to determine in the kit. Several types of air sprayers were evaluated for this reason. Table 4-7 outlines the particular spray devices used, as well as some specific spray, scrub and rinse times found to be useful in actual trials with **Decon-Check** to process whole protective suits.

Table 4-7. Whole Suit Timed Spray Tests II

	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt®Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Homax 4605P Air Sprayer
Spray Time (Full Suit Coverage)	1 min. 57 sec	1 min. 20 sec	1 min. 7 sec	1 min, 11 sec	1 min, 20 sec	1 min. 20 sec
Scrub Time	1 min. 46 sec	1 min. 36 sec	1 min. 25 sec	1 min, 21 sec	1 min, 20 sec	1 min, 20
Rinse Time	1 min. 11 sec	0 min. 48 sec	1 min. 13 sec	0 min, 58 sec	1 min, 5 se	1 min, 55 sec
Total Elapsed Time	3 min. 54 sec	3 min. 44 sec	3 min. 45 sec	3 min, 10 sec	3 min, 45 sec	3 min, 45 sec
Amount of composition (1:10 in water) Used (in cc) (Start/Finish)	1000/1700 (More Composition Added During Test)	2000/1400	2000/1200	1650/950	2000, 1200	2000,1200
Water Flow In Brush?	Yes	Yes	Yes	Yes	Yes	Yes
Average Height of Water in Tub After Rinse	1"	1"	1"	1"	1"	1"
Total Water Used (Scrub and Rinse)	28 gallons	21gallons	28 gallons	28 gallons	25 gallons	25 gallons

Test 1

Responder Class A protective suit, Model #50451, Serial #61807. Date manufactured May 25, 1993. Size – Large. Manufacturer – Lifeguard. NFPA 1991. Non-slippery as washed off compositions from treated garment.

Test 2

Same suit as in Test 1, plus Silver Flash Suit worn over the protective suit. Flashmax' #3, by Chemron, Inc., Order #56958. Aluminized oversuit/flash-fire cover suit.

Test 3/Test 4/Test 5/Test 6

Lifeguard Responder Class B suit, Model #80470, Serial #44651. Results: Foot wet after test 3 – possible leak. Slight orange color on socks. These results demonstrate the utility of the compositions as an aid in cleaning and/or deter-mining defects sites after decontamination.

Although the data in Table 4-7 indicates that the performance is quite similar for the various guns, other less tangible factors led to a decision to use the Homax 4605P spray unit. This spray gun has two outstanding performance factors not seen with the other units:

1. Spray overmist was greatly reduced, making product easier to apply; and,
2. Attachment of the prefilled **Decon-Check** container in "lock and load" fashion was much easier.

These important application factors favored the selection of the Homax delivery system.

4.3.2 Cleaning Efficacy

On a side by side comparison with common washing solution (DAWN®, 3 ounces diluted in 1 gallon water), **Decon-Check** removed a waxy grease contaminant from a Teflon® surface, while the washing solution provided only partial removal after extended scrubbing/rubbing. **Decon-Check** also provides a method for enhancing the adherence and visibility of a cleaning agent on a surface.

4.3.3 Decon-Check Kit Contents

The final **Decon-Check** Kit contents include:

- Four 2.2 liter bottles of **Decon-Check**
- One Homax 4605P Pneumatic Sprayer
- One Hose Attachable Brush Head
- One Extension Handle
- One Valve Assembly
- One Duffel Bag
- Instructions
- Instructional Video
- Material Safety Data Sheets

TRI/Austin is also offering a **Decon-Check** refill kit containing four 2.2 liter bottles.

4.3.4 Decon-Check First Article Field Evaluation

The first field trial of **Decon-Check** occurred at Dugway Proving Grounds (DPG) Dugway, Utah in January, 1995 concurrent with the penetrant evaluations. The basic composition of **Decon-Check** was briefly described, including the fact that it contains Limonene which gives it the citrus odor. General performance properties of **Decon-Check** were described, such as the ability to spray on a protective suit and have little if any vertical sag.

The protective suit that was evaluated was Chem-Fab 5800/6400 (STEPO) with 5 known wearings. **Decon-Check** was sprayed onto the inflated suit using a pneumatic sprayer. Generally comments were positive regarding the way in which **Decon-Check** sprayed and did not drain from the suit. DPG personnel seemed

surprised at how easily **Decon-Check** was removed when the rinsing/scrubbing step was performed.

DPG personnel suggested that a test be performed by removal of a grease contaminant from a butyl suit using **Decon-Check**. This was done and the results were positive as the grease was easily removed. Residual **Decon-Check** was removed by rinsing with tap water. No indication of fluorescence was observed after the rinsing step. Finally they suggested that **Decon-Check** might be a candidate decontaminating aid for some ongoing tests that are being performed at DPG by Lockheed.

4.3.5 Preproduction Kits

This task encompassed the fabrication of a minimum of 25 preproduction kits. These kits included improvements based on recommendations from first article kit evaluations. Additional items addressed during this task covered the planning of scale-up of kits of preproduction quantities and consultation with the subcontractor for supply of sufficient materials for incorporation into the preproduction kits. Other items that were developed under this task included labels, MSDS sheets, and promotional literature.

TRI/Austin also funded the production of 200 additional kits in multiple production lots. The remainder of these kits will be used for introduction to commercial clients and distributors. Orders for kits have already been received. The following list shows the 25 locations for delivery of the kits where further evaluation will occur. The kits will be distributed as they would be received by prospective clients.

Deliverable Recipient List – Decontamination Kits

Mr. Charles Warr
Commander
West Desert Test Center
STEDP-WD-C-CT Attn: Warr
Dugway Proving Ground
Dugway, Utah 84022

Mr. Matt Whipple
U.S. Army
Natick RD&E Center
SSCNC-IPS
Kansas St.
Natick, MA 01760-5019

Mr. Larry Gossage
Commander
Pine Bluff Chemical Activity
Attn. SCBPB-CMM (Gossage)
10020 Kabrich Circle
Pine Bluff, AR 71602-9500

Mr. David Douthat
U.S. Army Corps of Engineers
Huntsville Division
P.O. Box 1600
Attn: CEHND-OE
Huntsville, AL 35807

Mr. Ken Miller
Commander
U.S. Army Chemical School
Attn: ATZN-CMC-SP (Miller)
Fort McClellan, AL 36205-5020

Dr. Rich Knudsen
Biosafety Branch Chief
Center for Disease Control
4770 Buford Highway
Atlanta, GA 30341-3724

Dr. Jim Baker
US Army Edgewood RDE Center
Aberdeen Proving Ground, MD 21010-5423

Mr. Ngai Wong
U.S. Air Force Armstrong
Attn: USAF AL/CFBD, Bldg. E3234

Mr. Bruce A. Park, P.E.
Chief of Fire & Emergency Services
ATTN: DAIM-FDF-B
Alexandria, VA 22315-3800

DoD Fire Departments:

Fort Sill	Redstone Arsenal
Fort Bliss	Lonestar Arsenal
Fort Sam Houston	Corpus Christi Naval Air Station
Fort Hood	Fort Worth Naval Air Station
Fort Carson	Kelly AFB
Corpus Christi Army Depot	Tinker AFB
Pine Bluff Arsenal	Dyess AFB

Other:

US Drug Enforcement Agency	NASA Houston

Alternate Sites:

Barksdale AFB	Randolph AFB
Lackland AFB	Brooks AFB
Sheppard Air Force Base	

5.0 COMMERCIALIZATION AND MARKETING

TRI/Austin is currently pursuing various market segments for the penetrant and decontamination products developed during this project. These markets include both DoD, and other governmental agencies, and private sector parties.

5.1 Product Identification

TRI/Austin is aggressively marketing the products as they were developed during the Phase II effort. Two products have been identified as viable, marketable technologies. They are:

1. Spot Check Analysis Kit for detecting holes and other defects in protective clothing.
2. A Decontamination Aid for use during decontamination of
   i) All types of protective clothing
   ii) Equipment used in decontamination procedures

Test results indicate that a single product can be made that combines these two functions. This is an exciting result, and opens up broad marketability for this single product.

5.2 Advertising

For the broadest commercialization of the products, (including non-government customers) trade journals and catalogs will serve as important advertising targets. Examples of these type of publications are: *Environmental Solutions, Occupational Safety and Health, Fire Engineering, Safety and Protection* and *Occupational Hazards*. Conventions such as the National Safety Convention and the Fire Instruction Conference will also provide good advertising opportunities.

5.3 Key Customers

The following key customers are being targeted with product advertising. These divisions represent the major groups of current potential product users.

5.3.1 Government

1. Department of Defense
2. Department of Energy (National Labs, such as Oak Ridge and Los Alamos)

5.3.2 Commercial

1. Industrial Hazmat Teams
2. Manufacturers of Protective Clothing
3. Fire Departments
4. Other Users of Protective Clothing

Training is an important application of the product(s). Its importance cannot be overlooked, since training drills generally occur more frequently then actual hazardous material incidents.

5.4 Customer Databases

TRI/Austin has access to the proprietary customer database of TRI/Environmental, another subsidiary of TRI/International. It can be broken up into three databases (Total of 2043 customers):

1. Fire departments (1099)
2. Commercial (737)
3. Government (204)

These databases have been supplied to TRI/Austin in ASCII format that is convenient for use in mass mailings. This database will continue to be an invaluable resource for product advertising.

5.5 Manufacturing/Assembly/Distribution

TRI/Austin is currently assembling product kits for distribution. O.E.M. manufacturers are being utilized to make the bulk formulations. We have also arranged for independent sales and distribution to complement internal sales efforts.

Distributors have been supplied with the following information:

1. Product prospectus
2. Product samples
3. Availability information
4. Unit/bulk cost
5. Production capacity
6. Delivery information

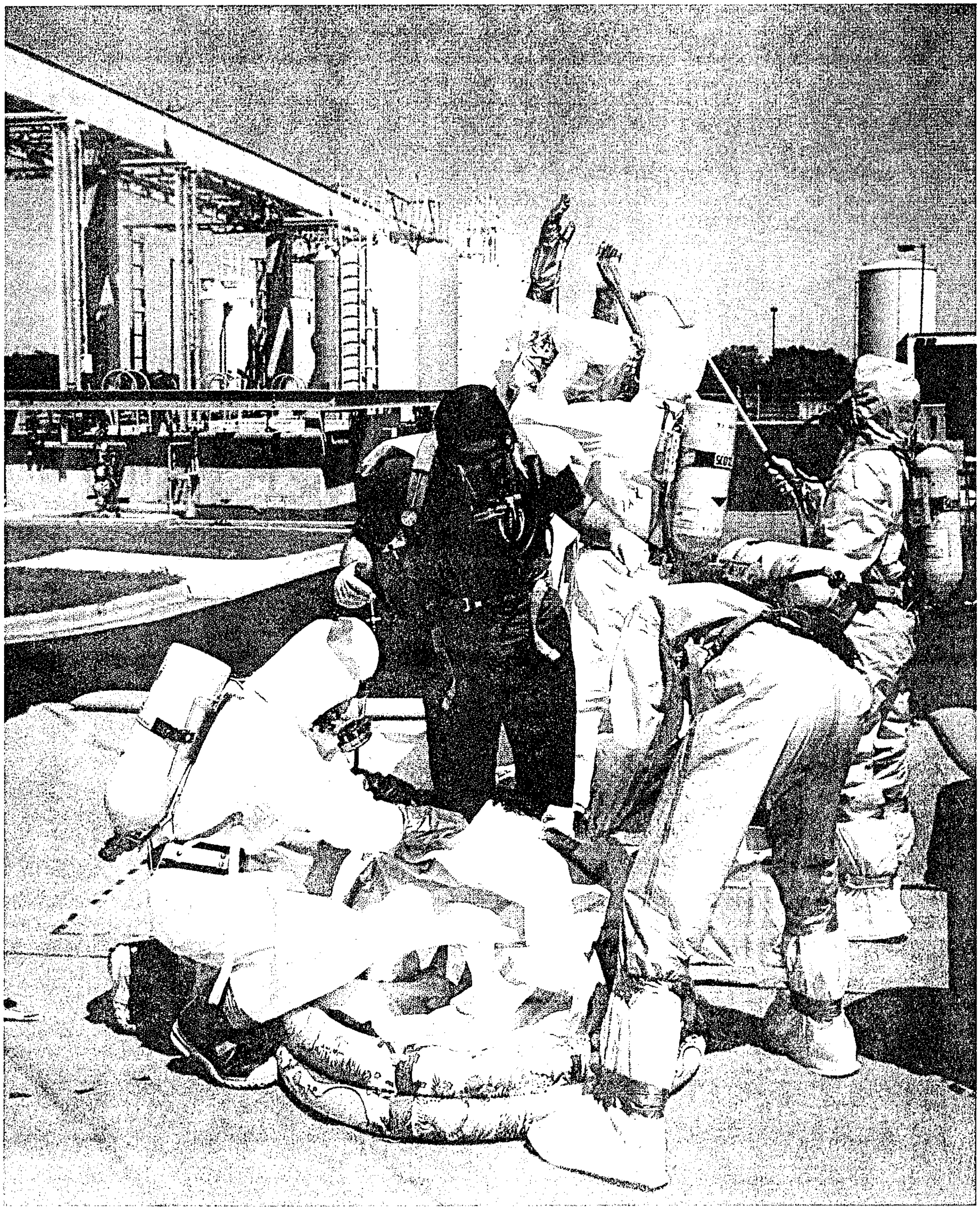
The list of distributors shown in Table 5-1 carry products for hazardous material handling and are possible distributors for our product(s). Other considerations for choice of distributors are size and geographical area coverage.

Table 5-1. Prospective Decon-Check Distributors

1. New Pig Corporation One Pork Ave. Tipton, PA 16684 800-HOT-HOGS	2. Ansul Fire Protection Spill Products Group One Stanton Street Marinette, WI54143 715-735-7411	3. Best Safety Supply Nat Street Milwaukee, WI 53225 414-827-8899
4. Field Safety 28 McGee Stamford, CT 06902 203-964-9199	5. Lab Safety Supply 3430 Palmer Drive Janesville, WI 53546 608-754-2345	6. Orr Safety Corporation 2360 Millers Lane P.O. Box 16-236 Louisville, KY 40216-0326 502-774-5791
7. Rice Safety Equipment Co. 4930 3rd Ave South Seattle, WA 98134 206-767-2524	8. Safeco, Inc. 435 E. Main Street, Suite 101 P.O. Box 28 Kingsport, TN 37662 615-378-5665	9. Safety Supply America 1 Civic Plaza Suite 320 Carson, CA 90745 213-513-0443
10. Vallen Corporation 13333 Northwest Freeway P.O. Box 3587 Houston, Texas 77253 713-462-8700	11. RMC Medical Darnel Road Philadelphia, PA 19154	12. Direct Safety Company 46th Street Dept. 5 Phoenix, AZ 85044 602-968-7009
13. Safety Supply America 2615 Homestead Place Box 4128 Rancho Dominguez, CA 90220800-772-6733	14. Mainstream Engineering Corporation 200 Yellow Place, Bldg. A Rockldge, FL 32955 800-866-3550	15. Safety Equipment Company Harney Road Box 31268 Tampa, FL 33631-3268 813-621-4921

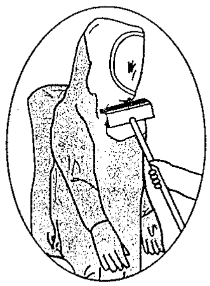
Appendix A
Formal Decon-Check Brochure

Miss a spot?



Standard Hazmat Decon Procedure

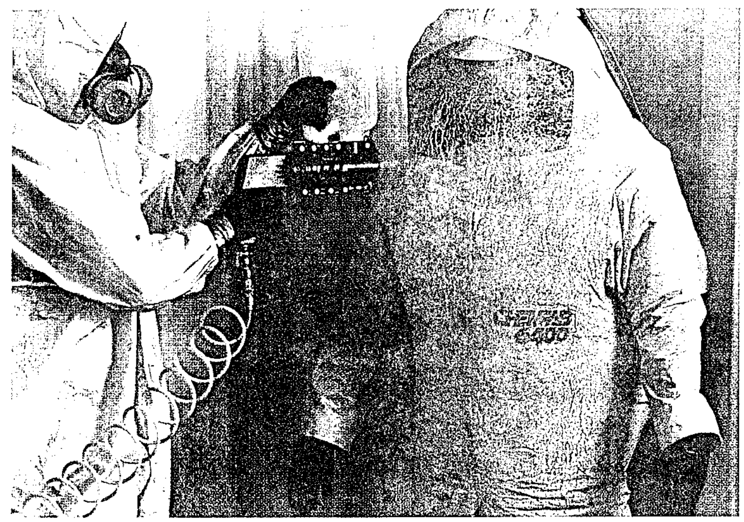
...Not with Decon-Check™ !



For the first time, it's possible to get a visual indication of cleaning effectiveness during decontamination procedures.

Decon-Check† is a spray-on/scrub-off protective clothing cleaner that:

- Includes a powerful detergent
- Has a bright color and
- Clings to most surfaces (removes easily with gentle scrubbing).



Spray On Decon-Check™

Decon-Check™ shows you where to scrub while its powerful detergent removes surface contaminants.

Scrub Contaminants Off

Decon-Check™ Liquid:

- Sprays on/Scrubs off – fast!
- Ready to use – No mixing required
- Works with a wide variety of protective clothing materials.

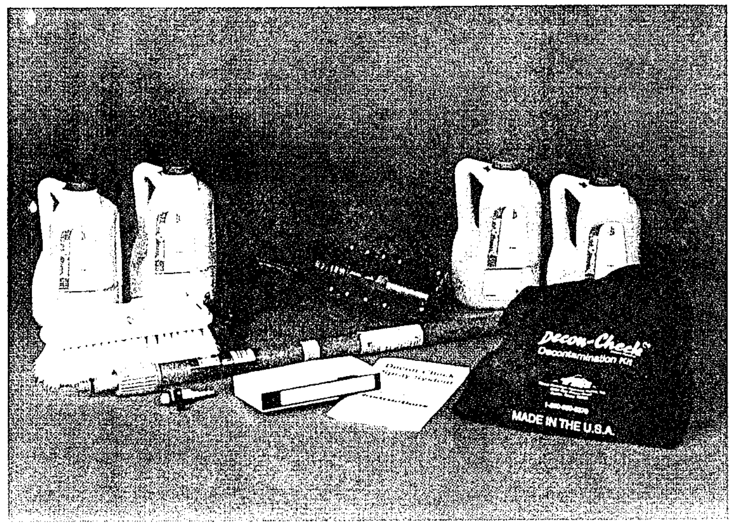
† – Patent pending

Decon-Check™ Spray System:

- Air powered sprayer uses SCBA or other compressed air source
- Easy "Lock and Load" bottle replacement
- Light, rugged, high efficiency sprayer.

Decon-Check™ Scrub Brush:

- Water-fed brush head
- Adjustable extension handle (2-4 feet) with water control valve



Decon-Check™ Starter Kit

Decon-Check™ is ideal for use in Hazmat:

- Training Drills
- Incident Response
- Site Remediation

Order Information

Part Number	Description
1000	Decon-Check™ Starter Kit Includes: Four 2.2 liter (2.3 quart) bottles of Decon-Check™ (enough to treat approximately eight suits) • Air Powered Sprayer • Brush Head • Extension Handle • Air Valve • Duffle Bag • Written Instructions • Instructional Video
1100	Decon-Check™ Refills Includes: Four 2.2 liter (2.3 quart) bottles (enough to treat approximately eight suits)

Replacement Parts

Part Number	Description
1010	Water-fed Brush Head
1020	Extension Handle (2'-4')
1030	Air Powered Sprayer
1040	Valve Assembly for air sprayer

When complete cleaning is important

Decon-Check™

Biodegradable • Non-Toxic • Non-flammable

Call toll free 1-800-880-8378 for ordering or technical information.

Decon-Check™ Sales Division
Texas Research Institute Austin, Inc.
A Texas Research International Company
9063 Bee Caves Road
Austin, Texas 78733-6201

1-800-880-8378 TEL
1-512-263-2101

1-512-263-3530 FAX

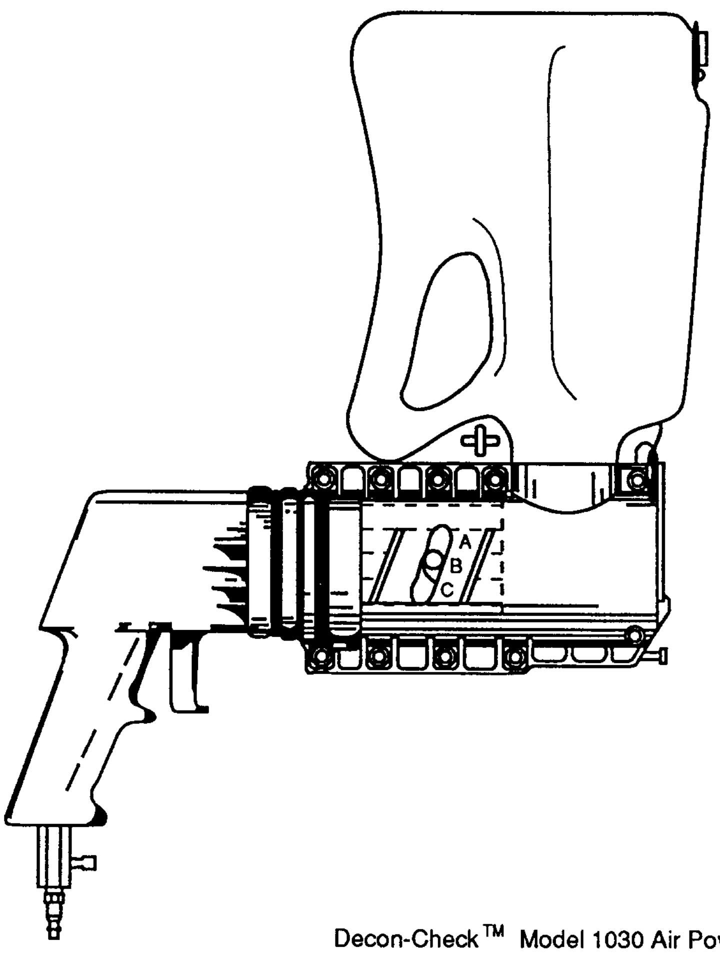
Distributed by:

Important: Read all instructions, warnings, and limited warranty before using Decon-Check™ products.

Appendix B
Decon-Check and Penetrant Instruction Manuals

Decon-Check™†

Decontamination Starter Kit
Instruction Manual



Decon-Check™ Model 1030 Air Powered Sprayer

ay Gun Made for TRI/Austin by HOMAX® Corporation
Patent Pending

Decon-Check™

Biodegradable • Non-toxic • Non-flammable

Congratulations! You have just purchased a revolutionary new product that gives you a visual indication of decontamination effectiveness during HazMat protective clothing decontamination procedures.

Decon-Check™ is a spray on/scrub off protective clothing cleaner that:

- Includes a powerful detergent,
- Has a bright color, and
- Clings to most surfaces (removes easily with gentle scrubbing).

Decon-Check™ shows you where to scrub while its powerful detergent removes surface contaminants.

Product Features

Decon-Check™ Liquid:

- Sprays on/Scrubs off – fast!
- Is ready to use – No mixing required
- Works with a wide variety of protective clothing materials.

Decon-Check™ Air-powered Spray System:

- Uses SCBA (Self Contained Breathing Apparatus) or other compressed air source
- Incorporates an easy "Lock and Load" bottle replacement system
- Is a light, rugged, high-efficiency sprayer.

Decon-Check™ Scrub Brush has:

- Water-fed brush head
- Adjustable extension handle (2'-4') with water control valve.

Decon-Check™ is ideal for use in HazMat training drills, incident response, and site remediation.

Kit Contents

Your Decon-Check™ Starter Kit includes the following:

- Four 2.2 liter bottles of Decon-Check™
- Air-powered Spray Gun
- Brush Head
- Extension Handle (2'-4')
- Air Valve Assembly
- Duffle Bag
- Written Instructions
- Instructional Video

The duffle bag can be used to store and transport the kit contents.

Air Supply Requirements

The air-powered Decon-Check™ spray gun can be used with any compressed air source that can deliver a minimum of 2.5 CFM at 30 PSI. Most 3/4 HP or larger compressors will work, and SCBA or other compressed air bottles that have been fitted with a suitable pressure regulator can also be used. **30 PSI is recommended for best performance of the system. Do not allow the spray gun handle inlet pressure to exceed 60 PSI.**

Note: An air shutoff valve assembly has been provided to conserve air when an SCBA or other bottled air source is used. The air flow should be turned on only when spraying Decon-Check™ fluid.

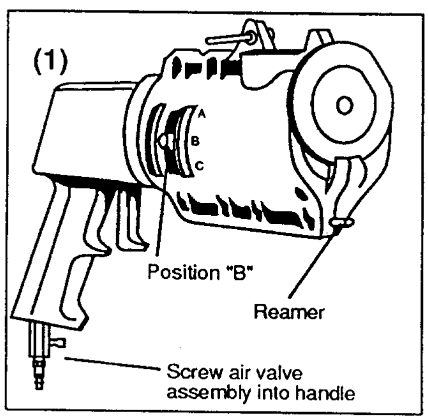
Instructions for Use

1. ***Important: Read all instructions, warnings, and material safety data sheets** before attempting to use Decon-Check™.*

Warning: The user must determine the suitability of this product for the decontamination task.

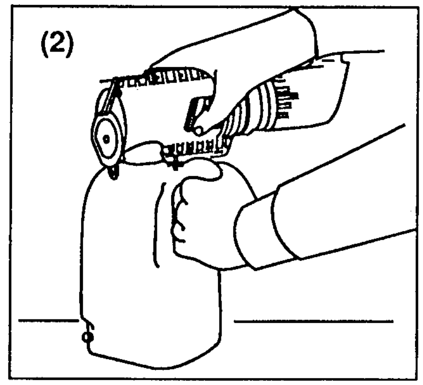
2. ***Install brush head on extension arm.*** Extend handle and tighten blue collar. Grip silver tube and firmly tighten brush head.

3. ***Adjust the spray gun.*** Insure that the spray gun selector switch is set to Position B. (See Figure 1.)



Instructions for Use (cont'd)

4. ***Open the Bottle.*** Place a Decon-Check™ bottle on a flat surface. Gently remove the cap.

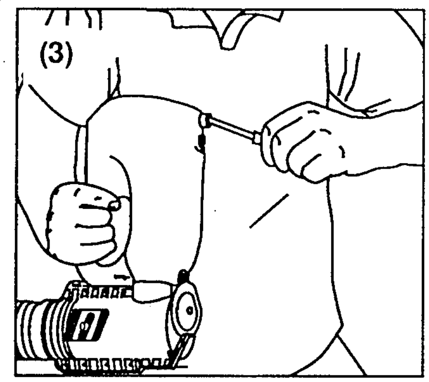


5. ***Attach the spray gun to the bottle.*** Hold the bottle firmly. Invert the spray gun and snap it onto the neck of the bottle. (See Figure 2). Rotate the spray gun until the locking tab passes through the slot in the bottle. Lock the bottle into place by turning the locking tab.

6. ***Vent the bottle.*** Important: This step must be performed to prevent over-pressurization which could cause injury.

   - ***Open the vent cap.*** The vent cap will snap back, revealing the vent hole area.

   - ***Pierce the vent hole.*** See Figure 3.

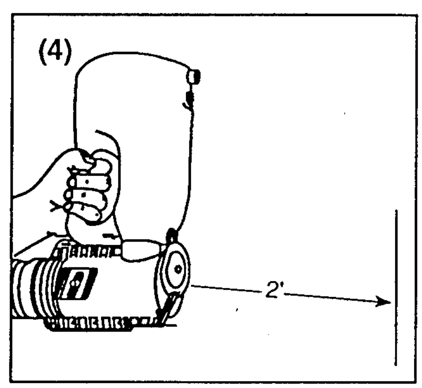


7. ***Reduce the compressed air pressure to 30 PSI.*** It is important to reduce the pressure before attaching the air source to the sprayer. 30 PSI is recommended for the best performance of the system.

⚠ **Warning**: Never let the air pressure exceed 60 PSI to avoid possible injury.

8. ***Open Air Valve.*** Opening the valve at the base of the spray gun handle prepares you for spraying.

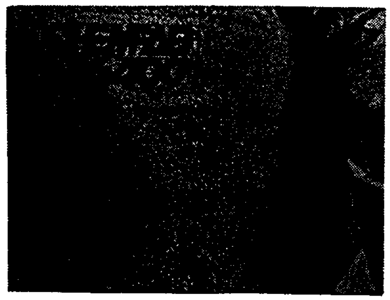
9. ***Spraying.*** Position the spray gun approximately 2 feet from suit and pull the trigger to spray (Figure 4). Move the spray gun parallel to the surface similar to spraying paint. **A LIGHT COAT IS ALL THAT IS NECESSARY** (see Figure 5). Continue to spray and cover the entire suit of protective clothing. The bright color of Decon-Check™ will help make sure that the entire protective clothing surface is covered.



If spraying upward, remember that Decon-Check™ is gravity-feeding into the spray gun. Hold the spray gun at 40°, which still allows material to flow into the chamber (see Figure 6).

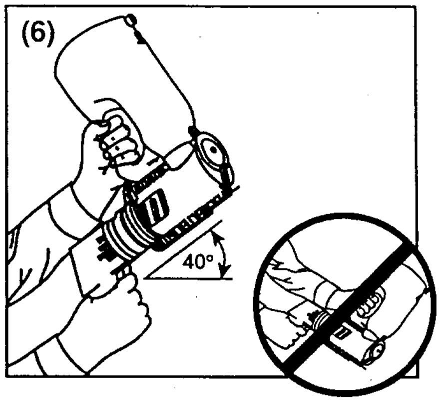
Important: Do not point the sprayer downward during use! (See Figure 6 inset.) This prevents wasteful loss of your Decon-Check™ fluid through the vent hole.

(5) View video for more details



9. ***Stop spraying.*** Release the trigger and turn off the air. After the air is turned off, pull the trigger to release any captured air from the system. When the bottle of Decon-Check™ fluid is empty, remove and replace it.

(6)



10. ***Scrub the protective clothing thoroughly.*** Adjust the water control valve on the extension handle to provide a slow, light flow of water to the brush head. Remove Decon-Check™ by scrubbing with the water-fed brush provided. The bright color of Decon-Check™ will help you distinguish areas that need to be scrubbed.

⚠ ***Warning**: The runoff solution may contain chemical contamination which could cause serious illness, injury or death. Collect the runoff solution in a portable pool or other liquid containment device. It should be treated as hazardous waste and disposed of in accordance with all federal, state, and local regulations.

11. ***Rinse.*** Increase the flow of water to the brush head. A small stream of water sprayed from the brush head can be used to remove any remaining traces of Decon-Check™. Collect and dispose of the runoff solution as previously described in Step 10 "*Warning".

12. ***Inspect the suit.*** Insure that all traces of Decon-Check™ have been removed. Bright color indicates incomplete cleaning. Scrub/rinse as needed to remove any remaining traces.

Instructions for Use (cont'd)

14. ***Further inspect the clothing*** to determine that no Decon-Check™ remain: Reuse of protective clothing and spray gear must be determined by the on-sit supervisor, safety officer, or other designated official.

15. ***Dispose of Contaminated Equipment.*** After spraying is completed, the scru brush and extension handle should be treated as hazardous waste and dispose of appropriately.

⚠ **Warning**: If the Decon-Check™ spray gun, air line and associated equipme have become contaminated, they should be disposed of in accordance wit appropriate regulations. If they are to be reused, they should be thorough rinsed with water and should be treated as hazardous materials and handle appropriately. Always use Decon-Check™ replacement parts. Replaceme brushes, extension handles and spray guns can be obtained from TRI/Austin c its distributors.

Safety Instructions

1. Only trained hazardous material team members should use this product. Training requirements are specified in 29 CFR, 1910.120.

2. Read warnings, instructions, and material safety data sheet (MSDS). The user must determine the suitability of this product for the decontamination task.

3. Inspect and analyze all protective clothing before reuse. Cleaning with this product may not remove all surface contamination. Decon-Check™ cannot remove matrix contamination that may be absorbed in the material. Refer to protective clothing manufacturers' instructions.

4. Items that come in contact with this product should be washed as soon as possible to reduce the likelihood of permanent staining.

5. Dispose of rinse solution, container, and other contaminated gear in accordance with state, local, and federal regulations.

6. Store product in a safe manner. Do not store below 32°F (0°C), or product may freeze.

7. Use only Decon-Check™ replacement parts.

Cleaning Your Decon-Check™ Spray Gun

⚠**Warning**: The Decon-Check™ spray gun should be cleaned only if it has not been contaminated with hazardous waste. If the spray gun has been contaminated, see Step 14, *Dispose of Contaminated Equipment*.

Cleaning Instructions

Rinse chamber of spray gun using warm water and a soft bristle brush. Continue until all traces of Decon-Check™ are removed.

Troubleshooting

***Problem*: Decon-Check™ liquid does not spray or atomize:**

- Is vent hole punctured and open?
- Use reamer to clear nozzle hole. (See Figure 1)
- If dried Decon-Check™ is keeping nozzle from opening, clean with water and a soft bristle brush.

***Problem*: Gobs or spits at nozzle:**

- Clean nozzle area.
- Reduce pressure (30 PSI is recommended for best results).

***Problem*: Decon-Check™ liquid drips/runs after spraying:**

- You may be applying too thick a coat.
- You may be spraying too close to the surface.

***Problem*: Spray is not fine enough:**

The pressure may be too low. 30 PSI is recommended for best results. **Never let the pressure exceed 60 PSI.**

Things to Remember

Keep bottle vent hole unclogged when spraying. **WARNING: If blockage occurs, immediately turn off air supply to prevent possible injury.**

Keep nozzle hole clear by using reamer supplied.

⚠**Warning**: Decon-Check™ is only an aid to the washing process. It does not render hazardous materials harmless and may not remove all surface contaminants. Decon-Check™ cannot remove matrix contaminants that may be absorbed into the material. Reuse of protective clothing and spray gear must be determined by the on-site supervisor, safety officer, or other designated official.

Decon-Check™

Biodegradable • Non-toxic • Non-flammable
Call toll free 1-800-973-3266 for ordering or technical information

Decon-Check™ Sales Division
TEXAS RESEARCH INSTITUTE AUSTIN, INC. (TRI/Austin)
A Texas Research International Company
9063 Bee Caves Road
Austin, Texas 78733-6201

1-800-97-DECON
1-800-973-3266
1-512-263-2101

1-512-263-3530 FAX

NOTICE AND LIMITED WARRANTY

It is the user's responsibility to determine the suitability of Decon-Check™ for intended use and the user assumes all risks arising from any nonsuitability of this product. The maker and sellers of Decon-Check™ warrant Decon-Check™ and its applicator parts to be free from defects in materials and workmanship when used in accordance with the warnings and instructions which accompany the product. Should there be a proven defect in the product, the obligation of the maker and sellers of Decon-Check™ under this warranty is limited solely to replacement of the product; all other liability for damages or loss, including consequential damages, is excluded. THIS WARRANTY IS GIVEN EXPRESSLY AND IN PLACE OF ALL OTHER EXPRESS OR IMPLIED WARRANTIES, INCLUDING THE WARRANTY OF MERCHANTABILITY AND FITNESS FOR PARTICULAR PURPOSE.

TRI/AUSTIN, INC.
A Texas Research International Company
9063 Bee Caves Road
Austin, Texas 78733-6201

1-800-97-DECON
1-800-973-3266

D219.2
Rev. 7/95

CHEMICAL PROTECTIVE CLOTHING
LIQUID PENETRANT INSPECTION
Kit Contents List and Procedures

TEXAS RESEARCH INSTITUTE AUSTIN, INC.
Materials Division
9063 Bee Caves Road
Austin, Texas 78733-6201
(512) 263-2101

D202.7

TABLE OF CONTENTS

First Article Penetrant Spot Test Kit Contents

1. UV Light
2. Battery Pack
3. Battery Pack Accessories
4. UV Glasses
5. Funnel
6. Wipes (1 Box)
7. 4 Pair Gloves
8. 1 Apron
9. 1 Qt. TRI-C Penetrant
10. $^1/_4$ Pint Dauber Can with TRI-C Penetrant
11. Small Pump Spray Bottle (Red) for Water
12. 2 Cans KO-19 Remover
13. 1 Large Spray Bottle (Gray)
14. 2 China Markers
15. 2 Cans DR-60 Cleaner/Remover
16. Water Hose Nozzle
17. Scrub Brush
18. 2" Paint Brush
19. MSDS Sheets

Liquid Penetrant Inspection Procedure

Test Temperature: The temperature of the penetrant material and the surface of the test sample to be examined should be between 40° and 120°F.

Surface Condition: All surfaces to be examined must be free of any grease, paint, oily films, dirt, etc., that might interfere with ingression of the penetrant.

Precleaning: Clean grease, oily residues, etc., from the area to be inspected using Sherwin DR-60 Cleaner/Remover. The inspection area should be thoroughly dry prior to further processing.

Figure 1 – Precleaning Inspection Area

)rying of Inspection Area: After precleaning, the inspection area must be ıllowed to dry for a minimum of 5 minutes by normal evaporation or the ıpplication of warm air, (less than 120°F).

›enetrant Application: After agitation of the penetrant solution, apply a beral amount of fluorescent penetrant to the area to be inspected using the upplied dauber applicator. Allow the penetrant to dwell for 10 minutes on ıe inspection area. The examination surface should be kept wet with ›enetrant during the prescribed dwell time and not allowed to dry out.

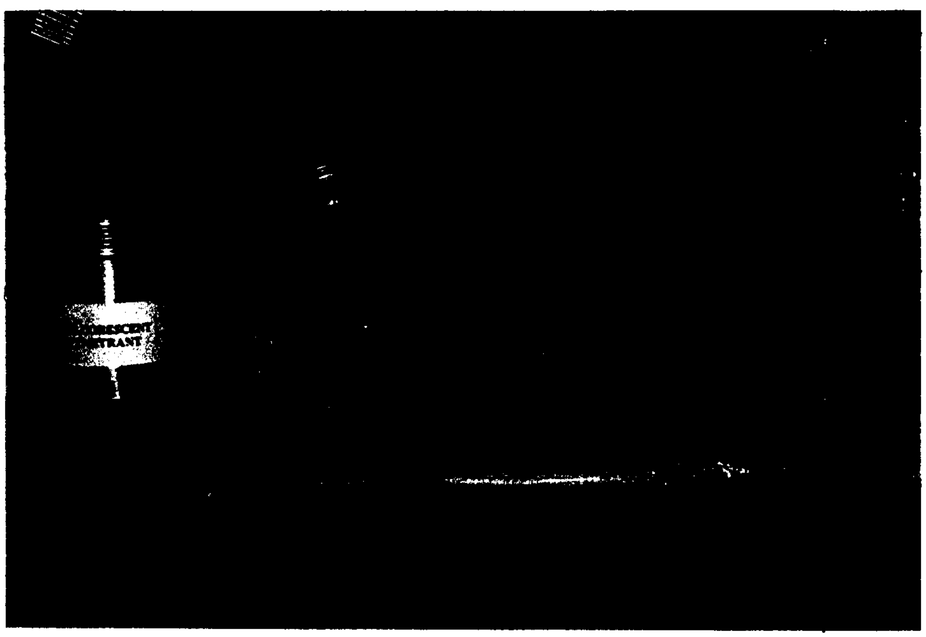


Figure 2 – Penetrant Application

Removal of Excess Penetrant: After the required penetration dwell time, remove the excess penetrant using one of the wipe towels supplied in the test kit. Apply KO-19 remover to inspection area. Allow KO-19 to dwell for at least 1 minute. Rinse KO-19 off with water using the supplied hose nozzle or spray bottles. Allow area to dry as outlined above in "Drying of Inspection Area."

Inspection: Perform all Inspections in a darkened area using the supplied UV light. **SAFETY WARNING:** Do not look directly into UV light and always wear supplied UV glasses.

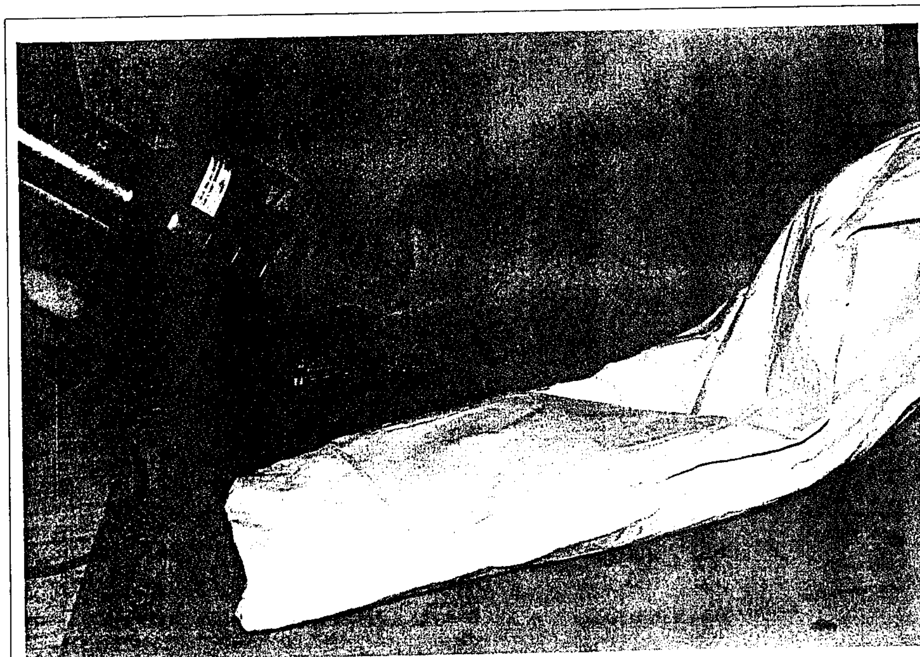


Figure 3 – Spot Inspection with UV Light

Identification of Defects: In order to detect defects on the protective clothing material it may be necessary to turn the suit inside out. Doing so allows the observance of fluorescent "halos" around the defect area. These "halos" can be $^1/_4$ to 4 inches in diameter depending on the defect size. Figure 4 shows the penetrant process and how it results in halo formation. Figure 5 shows the halo effect on CP material with a battery powered UV light.

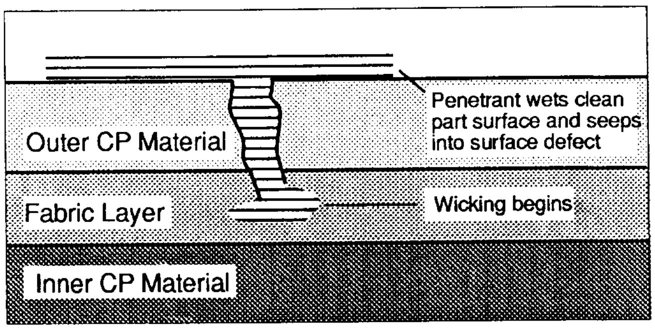


Figure 4a – Penetrant Process in CP Materials
Following precleaning, penetrant is applied to test surfaces and wicks into surface discontinuities during the penetrant dwell time. If crack depth exceeds outer CP material thickness, wicking begins immediately.

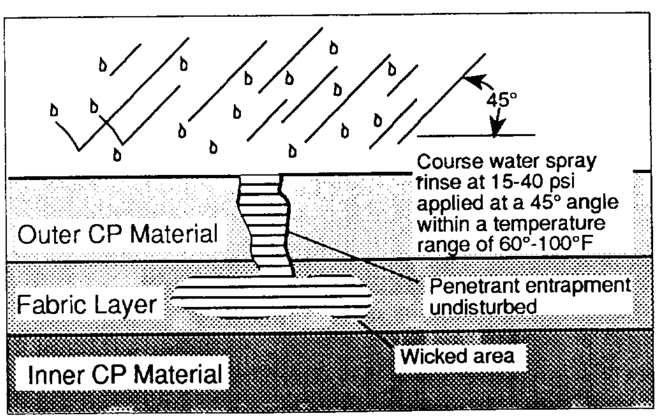


Figure 4b – Penetrant Process in CP Materials (cont'd)
Angled coarse water spray removes excess water-soluble penetrant from test surface, but does not remove entrapped penetrant.

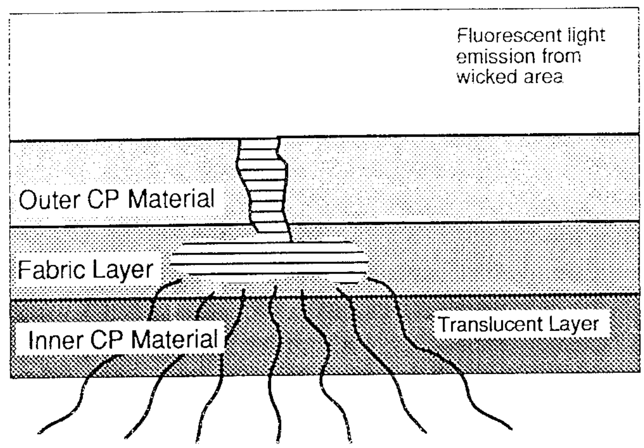


Figure 4c – Penetrant Process in CP Materials (cont'd)
Ultraviolet light excitation causes fluorescent penetrant indication to glow-in-the-dark by radiating through translucent layers.

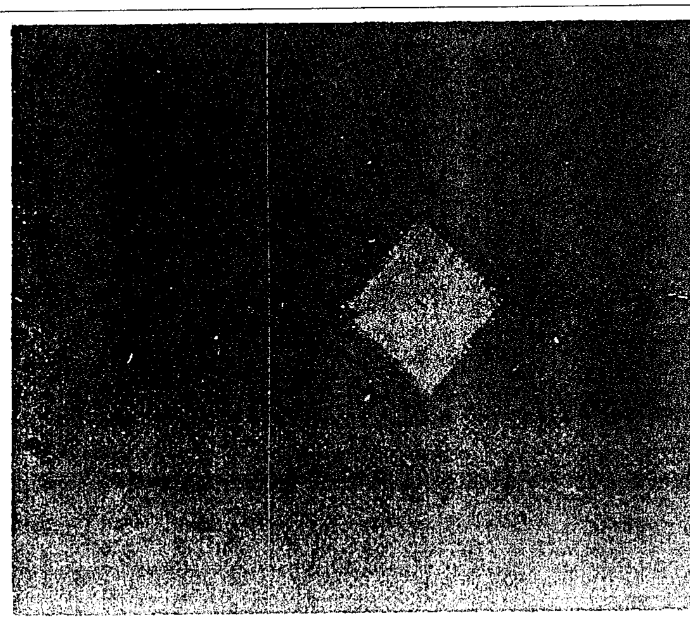


Figure 5 – Showing the Halo Effect with a Battery Powered UV Light Source